

EVALUATION OF THE CHEMICAL DURABILITY  
OF  
IOWA FLY ASH CONCRETES

PHASE I PROGRESS REPORT  
MARCH 31, 1991

IOWA DOT PROJECT HR-327  
ERI PROJECT 3295

Sponsored by the Highway Division of the  
Iowa Department of Transportation and the  
Iowa Highway Research Advisory Board

ENGINEERING RESEARCH INSTITUTE

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**EVALUATION OF THE CHEMICAL DURABILITY  
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**"The opinions, findings, and conclusions expressed in this publication are those of the authors and not necessarily those of the Highway Division of the Iowa Department of Transportation."**

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## INTRODUCTION

The following report summarizes research activities conducted on Iowa Department of Transportation Project HR-327, for the period April 1, 1990 through March 31, 1991. The purpose of this research project is to investigate how fly ash influences the chemical durability of portland cement based materials. The goal of this research is to utilize the empirical information obtained from laboratory testing to better estimate the durability of portland cement concrete pavements (with and without fly ash) subjected to chemical attack via the natural environment or the application of deicing salts.

This project is being jointly sponsored by the Iowa Department of Transportation and the Iowa Fly Ash Affiliate Research group. The research work is also being cooperatively conducted by Iowa State University and Iowa Department of Transportation research personnel. Researchers at Iowa State University are conducting the paste and mortar studies while Iowa Department of Transportation researchers are conducting the concrete study.

## RESEARCH APPROACH

### Sampling Scheme

Fly ashes from Council Bluffs (unit #3), Louisa, Port Neal (unit #4) and Ottumwa generating stations were selected to represent the range of Class C fly ashes available in Iowa. Fly ash from M.L. Kapp generating station (Clinton) was selected to represent the Class F fly ashes available in Iowa. The general locations and ash production rates of the various power plants are illustrated in Figure 1. Details concerning the various powerplants are summarized in Appendix A.

The fly ash sample from a given power plant was taken on a single day (i.e., the samples were not composite samples). Enough sample was taken from each power plant to approximately fill two 55 gallon drums (i.e., roughly 500 pounds of fly ash). The fly ash samples were then delivered to the Materials Analysis and Research Laboratory (MARL) at Iowa State University where they were sub-sampled, labeled and dated. The MARL personnel then delivered one barrel of each source of fly ash to the Iowa Department of Transportation (IDOT) Materials Laboratory for use in the concrete portion of the research project.

Three different sources of portland cement were chosen for use in the project. Two of the sources produced Type I portland cement, while the remaining source produced Type V (sulfate resistant) portland cement.

Neal 4  
100,000 tpy

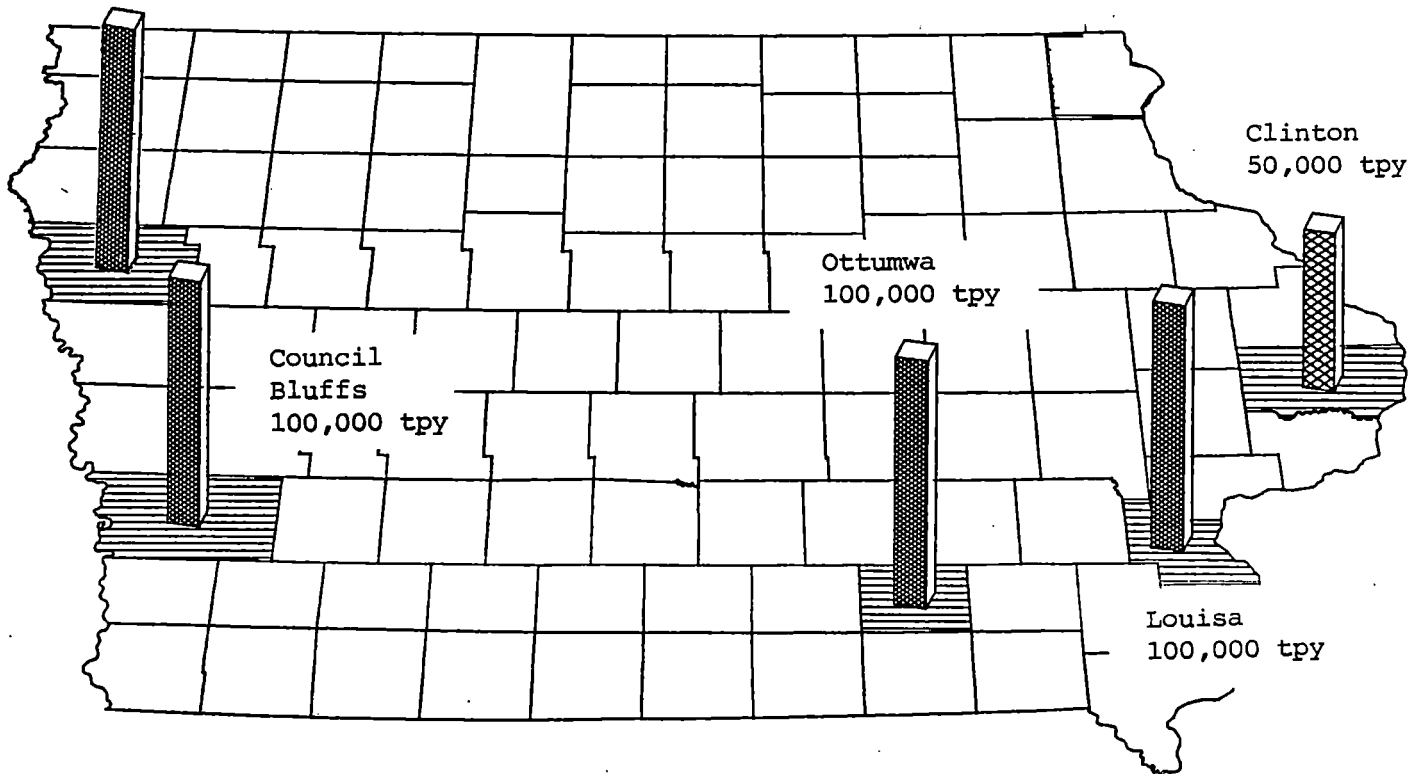


Figure 1. General location and amount of fly ash produced at the power plants studied in this project.

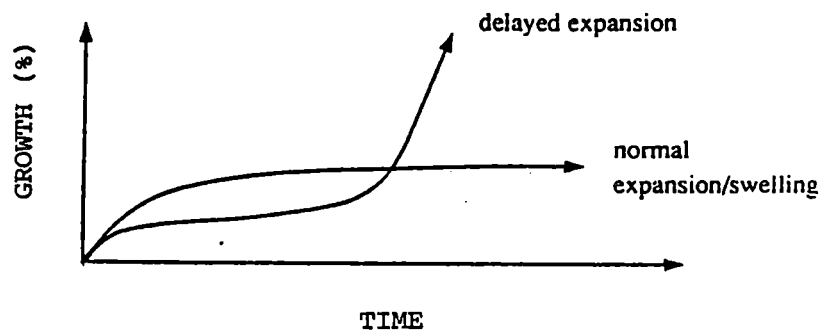


Figure 2. Diagram illustrating the response of test specimens to sulfate attack.

The Type I portland cements consisted of a low alkali cement (Dundee) from Mason City, Iowa, and a high alkali cement from Davenport, Iowa. The Type V cement was obtained from Rapid City, South Dakota. All of the cements were delivered to the laboratory in standard (94 lb) bags.

#### Testing Scheme for Sulfate Attack

Many different criteria can be used to estimate the sulfate resistance of portland cement products. For this research project specimen growth (or linear expansion) is the major property that is being used to evaluate the durability of portland cement-fly ash pastes, mortars and concretes immersed in sulfate bearing solutions. Concrete specimens are also being monitored for weight change and dynamic modulus of elasticity (sonic modulus). The typical response that is expected from any given specimen is illustrated in Figure 2. Failure can be defined as some predetermined value of growth or the experiment can be continued until the specimen physically disintegrates.

Concrete specimens for sulfate durability testing were prepared at the IDOT. The concrete mixes employed two sources of cement (Type I and Type V), four sources of fly ash (Council Bluffs, Louisa, Ottumwa and Clinton), and two different coarse aggregates (Jabens and Lamont). The fine aggregate used for the mixes was from near Bellevue, Iowa. All of the concrete mixes were proportioned using IDOT C-3 mix specifications. Fly ash was replaced for cement on a 1 to 1 basis throughout this study. Fly ash replacements of 7.5, 15 and 30% (by weight) were used in this study. Concrete beams with nominal dimensions of 4" x 4" x 18" were molded for sulfate durability testing. Two cylinders (4.5" x 9") were also molded from the mix to evaluate the 28 day compressive strength of the concrete. All of the beam specimens were moist cured for at least 28 days before immersion in the sulfate solution. Aqueous solutions with two different concentrations of sulfate were used in this study. The first solution contained 10%  $\text{Na}_2\text{SO}_4$  (by weight), this solution has been used by other researchers [1,2], and has proven to be quite aggressive to portland cement concretes. The second solution contained 10% mixed salt (by weight). The composition of the mixed salt was 95%  $\text{NaCl}$  and 5%  $\text{Na}_2\text{SO}_4$ , this was used to simulate a "worse case" deicing salt. Hence, the second solution ultimately contained 9.5%  $\text{NaCl}$  and 0.5% of  $\text{Na}_2\text{SO}_4$ . Technical grade (or better) purity  $\text{Na}_2\text{SO}_4$  and  $\text{NaCl}$  were used to make both solutions.

Mortar specimens for sulfate durability testing were prepared in accordance with ASTM C 1012-90 [3], with two notable exceptions. First, the accelerated curing method described in ASTM C 1012 was not used. Instead, specimens were demolded after 1 day of moist curing and then placed in saturated lime water until they reached a minimum strength of 2850 psi. The strength versus time relationship was established using 2 inch cube specimens that were molded, cured and tested as described in ASTM C 109 [4]. Secondly, only two specimens (rather than the four suggested by the ASTM) were molded for each mixture. Four different replacements of fly ash for cement (7.5, 15, 22.5 and 30%, by weight) were studied in this project.

The mortar specimens were subjected to aqueous solutions containing two different concentrations of sulfate. The first solution contained 5%  $\text{Na}_2\text{SO}_4$  (by weight). The second solution contained 9.5% NaCl and 0.25%  $\text{Na}_2\text{SO}_4$  (by weight). Reagent grade NaCl and  $\text{Na}_2\text{SO}_4$  were used in the mortar phase of this study. A tank containing lime water was used to assess the expansive potential of many of the mortar mixes.

Paste specimens for sulfate durability testing were mixed using a procedure developed in our earlier studies [5]. Since paste specimens are homogenous on a small scale a preliminary experiment was performed to see if small cylindrical test specimens (13/16" diameter by 3" long) could be substituted for the larger (1" x 1" x 11.25") prismatic specimens. Only one concentration of sulfate solution (5%  $\text{Na}_2\text{SO}_4$ ) was used to assess durability of the paste specimens. Pending the results of this preliminary study a detailed study will be made of the durability of fly ash cement pastes.

#### Testing Scheme for Alkali Attack

Mortar specimens for alkali attack were made in accordance with ASTM C 311-90[4]. This study used two Type I portland cements, all five of the fly ash samples mentioned earlier in this report, and three different fine aggregates (pyrex glass, standard ASTM C 109 sand and a Class V aggregate from Oreapolis, IA). Five different levels of fly ash replacement (7.5, 15, 22.5, 30 and 50%) were used in this research project. This should give a clear indication of the presence of a pessimum amount of fly ash.

#### Chemical Testing Scheme

All of the raw materials were subjected to chemical tests. Typically, x-ray analysis was used to define both the bulk composition and the minerals present in a given material.

X-ray diffraction (XRD) was used to identify the major and minor crystalline constituents present in each material. A Siemens D 500 X-ray diffractometer was used throughout this study. The diffractometer was controlled by a PDP 11/23 computer via an LC500 interface. A copper x-ray tube was used for all diffraction work. The diffractometer was equipped with a diffracted beam monochromator and medium resolution slits.

X-ray fluorescence (XRF) analysis was used to quantify the major, minor and selected trace elements in the various materials. A Siemens SRS 200 sequential x-ray spectrometer was used for all of the analyses. The spectrometer was fully computer controlled. A chrome x-ray tube was used throughout the study.

A Beckman DU-2 flame photometer was used to determine the available alkali content (Na and K expressed as equivalent  $\text{Na}_2\text{O}$ ) of the various fly ashes. An oxygen-hydrogen flame was used for all analyses.

#### CURRENT STATUS

##### Chemical Tests

The primary chemical tests have been completed for the five fly ashes, three cements, coarse aggregates, fine aggregate, pyrex glass and reagent and technical grade chemicals used during the first year of the project. Specific mortar specimens have been subjected to XRD analysis to help in pinpointing the potential cause of expansion in the various specimens. Paste specimens have not yet reached failure so they have not been subjected to XRD analysis.

##### Physical Tests

All of the ASTM C 1012 (sulfate durability) mortar specimens have been molded and are currently immersed in solutions containing sulfates or lime water. Also, a series of mortar specimens were made to assess the influence of curing time on the sulfate durability test (ASTM C 1012). All of the specimens have been molded and are currently immersed in a 5%  $\text{Na}_2\text{SO}_4$  solution.

The preliminary series of paste specimens have also been molded and are currently immersed in sulfate bearing solutions. Continued work with additional fly ash-cement paste specimens will depend on the outcome of this preliminary experiment.

A total of 52 concrete mixes have been completed by IDOT personnel. All of the concrete specimens were moist cured for at least 28 days before they were immersed in a sulfate bearing solution.



The preparation of mortar specimens for alkali attack has almost been completed. At present, about 60% of the mortar specimens have been molded and are presently being cured at 38°C. It is estimated that about one more month will be required to complete this task.

In summary, the project is proceeding well and the work is being performed on a schedule close to the one listed in the research proposal.

## PRELIMINARY RESULTS AND DISCUSSION

### Chemical Tests

All of the materials used in this project have been subjected to chemical testing. However, due to the potential for sampling error in any bulk chemical testing scheme these results must only be considered as the current best estimates for the composition of the materials used in this study. Additional samples of the various materials are still being analyzed and the test results listed in this report may change as new test results become available. However, at this time we do not anticipate any major changes to these preliminary results.

The chemical compositions of the five fly ash samples are listed in Table I. The fly ashes were also subjected to a physical testing program similar to that suggested in ASTM C 618. The results of the physical testing program are listed in Appendix A. Overall, the five fly ashes chosen for the research program exhibited a good range of chemical and physical properties.

X-ray diffractograms of the five fly ashes are shown in Figures 3 through 7. The compounds identified in the various diffractograms are summarized in Table II. Please note that some of the compounds listed in Table II are not directly evident in the diffractograms of the bulk fly ashes; however, additional treatments (i.e., acid extraction, particle size separation, etc.) were used to verify the presence of the various compounds.

Currently it is not possible to accurately estimate the amounts of the various compounds present in the fly ashes. However, due to the influence of tricalcium aluminate on sulfate attack, the amount of tricalcium aluminate present in each fly ash was estimated using quantitative x-ray diffraction. The tricalcium aluminate estimate was made by spiking the raw fly ash samples with known amounts of pure tricalcium aluminate (cubic structure, XRD pattern matched JCPDS#38-1429). Note, that all of the fly ash samples had tricalcium aluminate contents of less than 10% (by weight).

Table I  
Summary of Bulk Chemistry of the Fly Ash Samples

	Sample ----->				
<u>Oxide (wt%)</u>	<u>Clinton</u>	<u>Louisa</u>	<u>Ottumwa</u>	<u>Neal 4</u>	<u>Council Bluffs</u>
SiO <sub>2</sub>	51.1	35.9	30.9	35.1	30.8
Al <sub>2</sub> O <sub>3</sub>	18.4	22.8	20.2	18.0	16.9
Fe <sub>2</sub> O <sub>3</sub> (T)	14.1	5.8	5.9	5.7	6.9
Sum:	83.6	64.5	57.0	58.8	54.6
SO <sub>3</sub>	1.8	1.5	2.7	3.2	4.0
CaO	5.0	23.2	26.3	26.6	29.1
MgO	1.2	4.4	4.8	4.9	6.5
P <sub>2</sub> O <sub>5</sub>	0.15	1.46	2.12	1.14	0.63
K <sub>2</sub> O	2.06	0.51	0.35	0.39	0.24
Na <sub>2</sub> O	0.6	1.4	2.5	1.6	2.3
TiO <sub>2</sub>	0.95	1.55	1.42	1.41	1.28
SrO	0.03	0.39	0.47	0.41	0.42
BaO	0.07	0.78	0.97	0.83	0.75
moisture content	0.12	0.01	0.05	0.08	0.05
Loss on Ignition	1.05	0.14	0.23	0.41	0.26
Available Alkali	0.91	1.28	2.08	0.95	1.68
Repeat	0.91	-	1.81	0.94	1.62
% Acid Soluble	12.7	69.6	75.8	70.3	74.4
ASTM Class	F	C	C	C	C
Tricalcium Aluminate (C <sub>3</sub> A, wt%)	ND	4.5	6.5	6.0	7.7

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ND - not detected

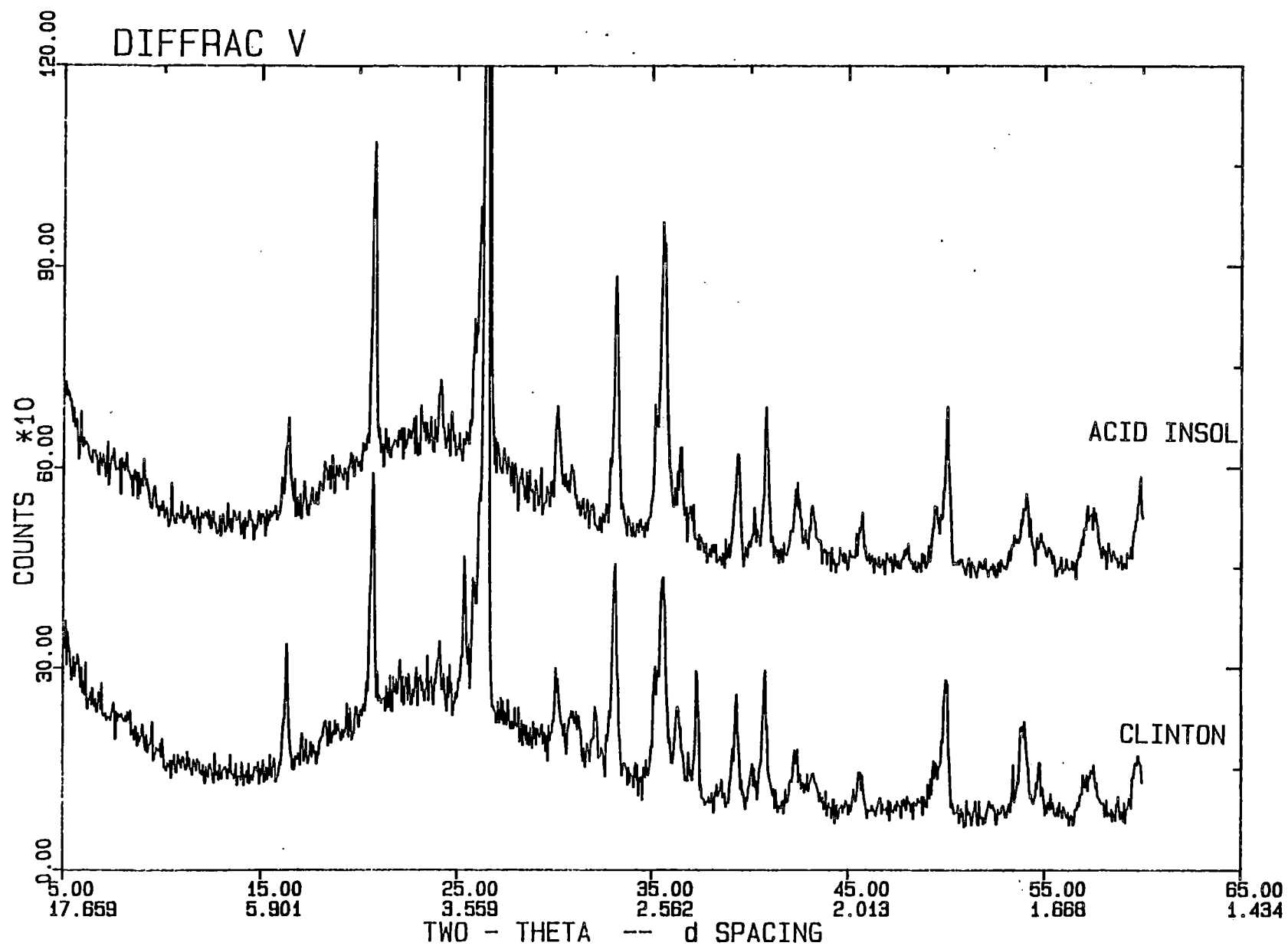


Figure 3. X-ray diffractogram of Clinton fly ash.

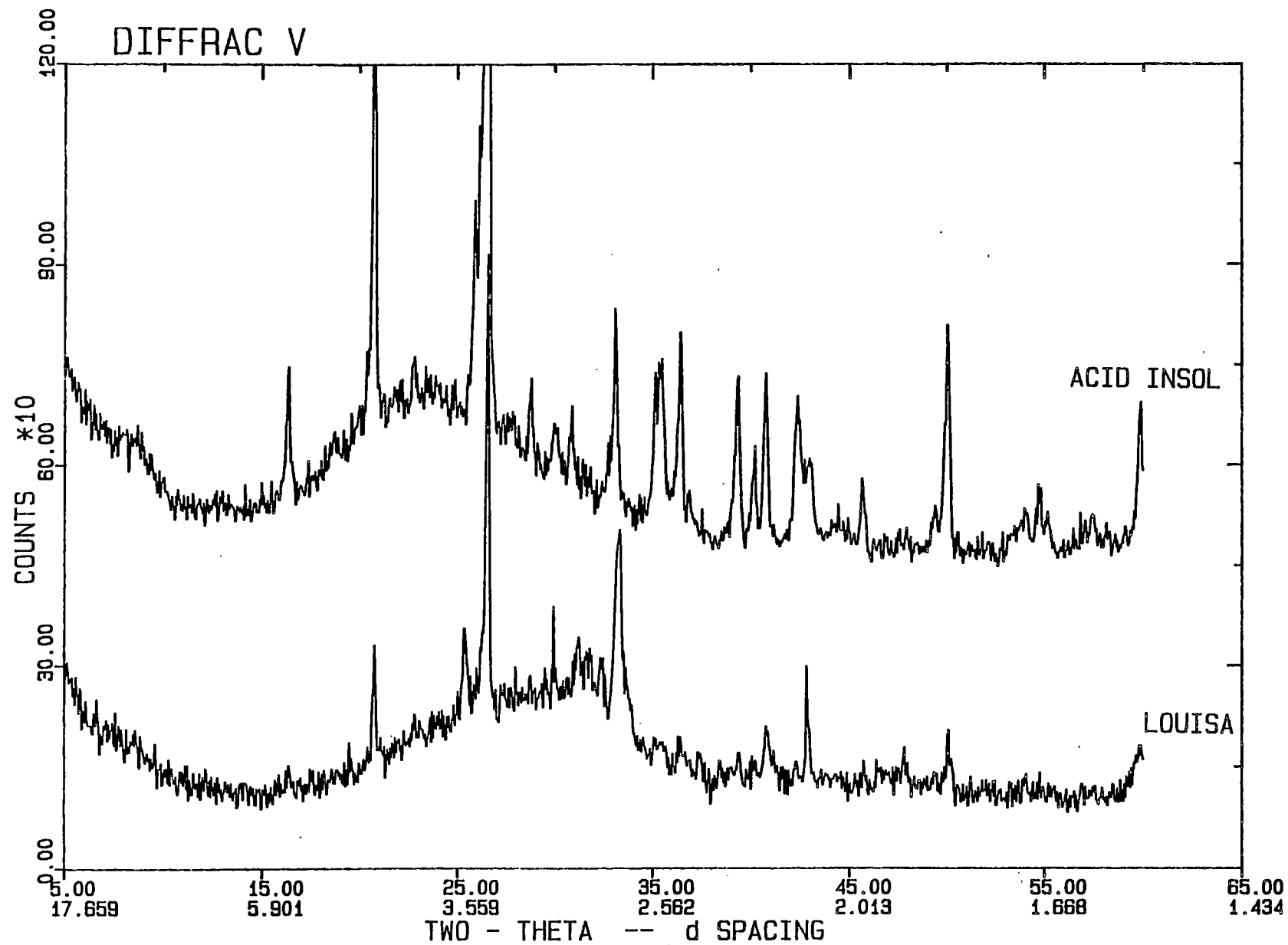


Figure 4. X-ray diffractogram of Louisa fly ash.

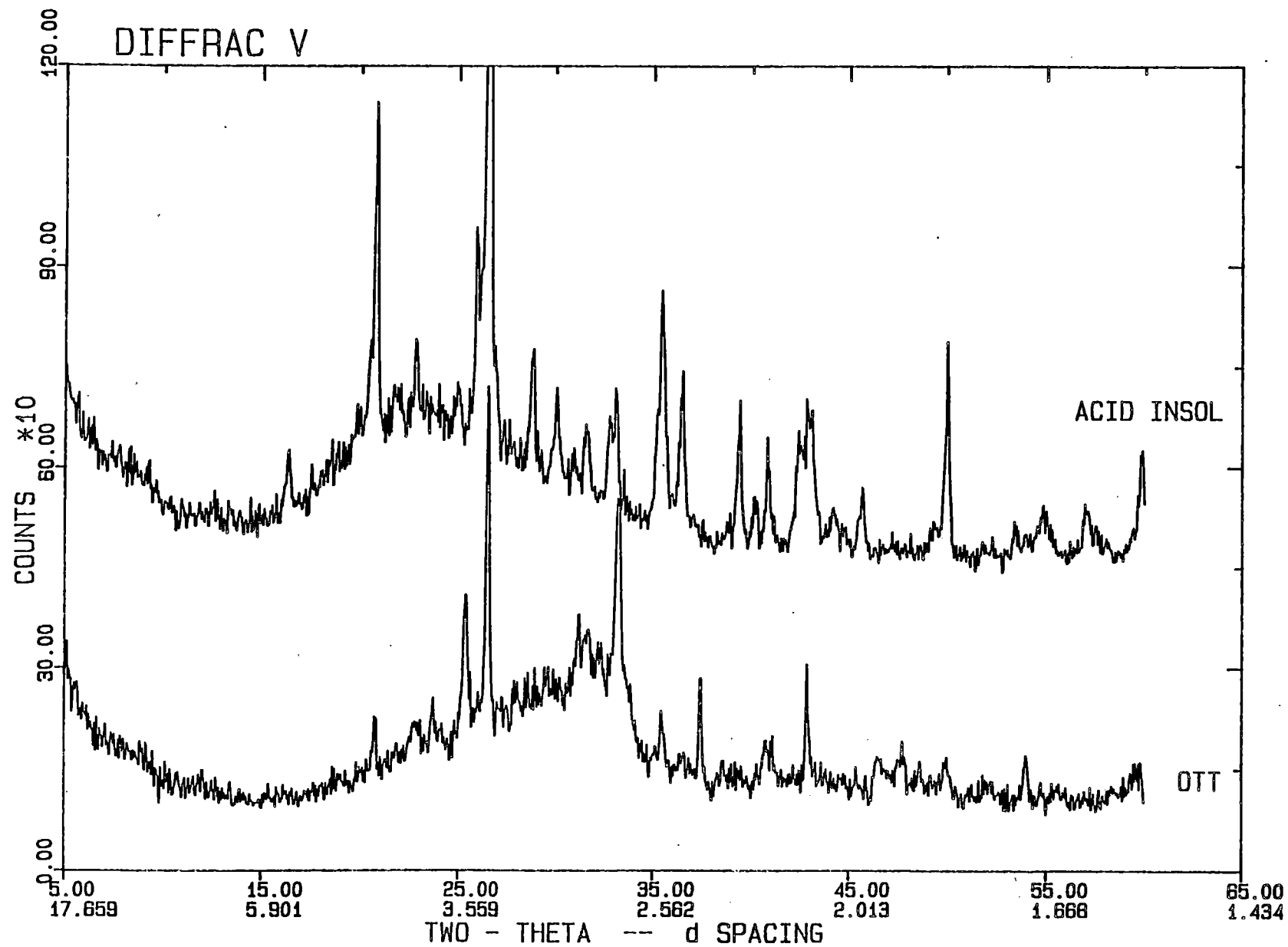


Figure 5. X-ray diffractogram of Ottumwa fly ash.

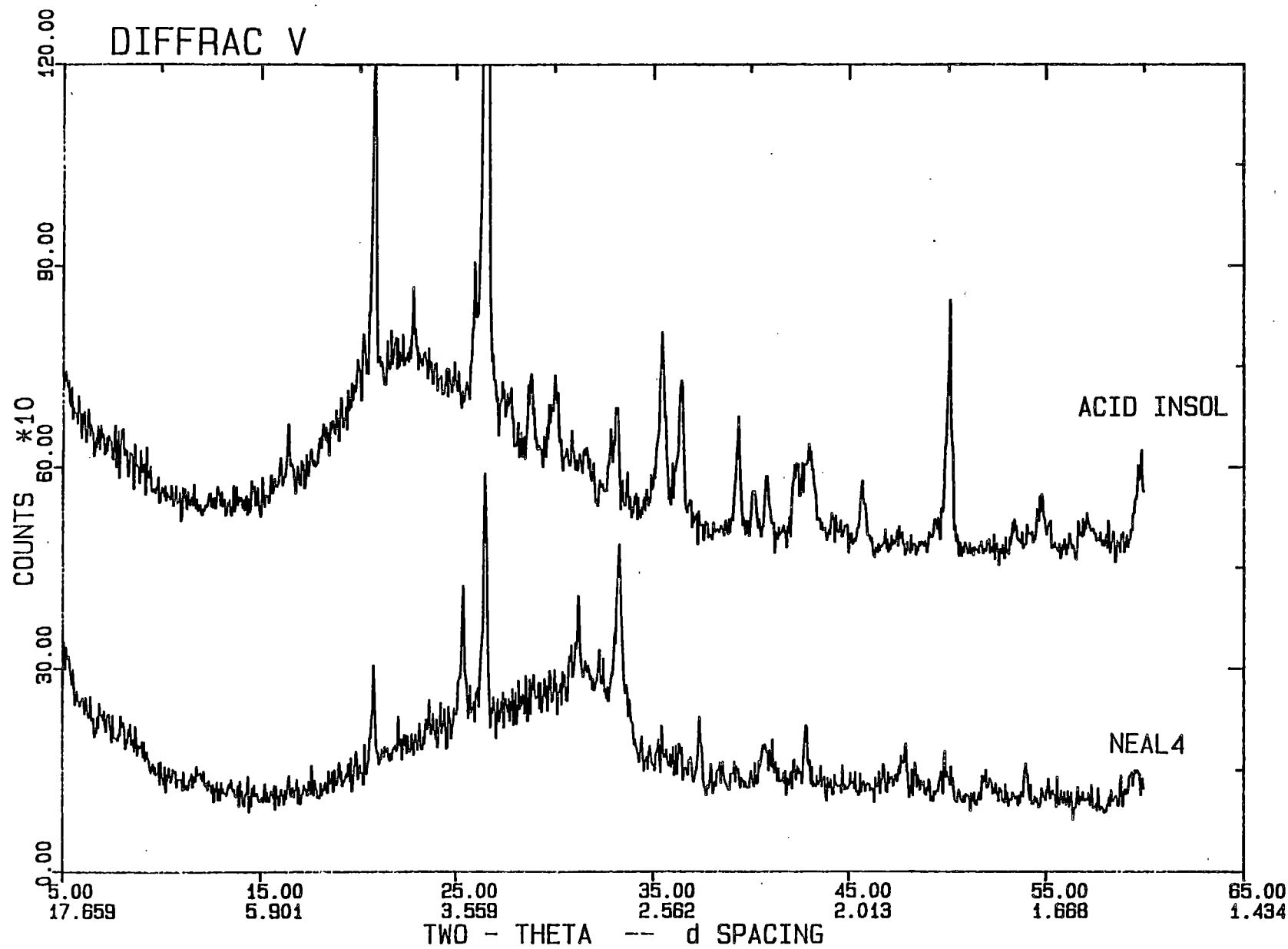


Figure 6. X-ray diffractogram of Neal 4 fly ash.

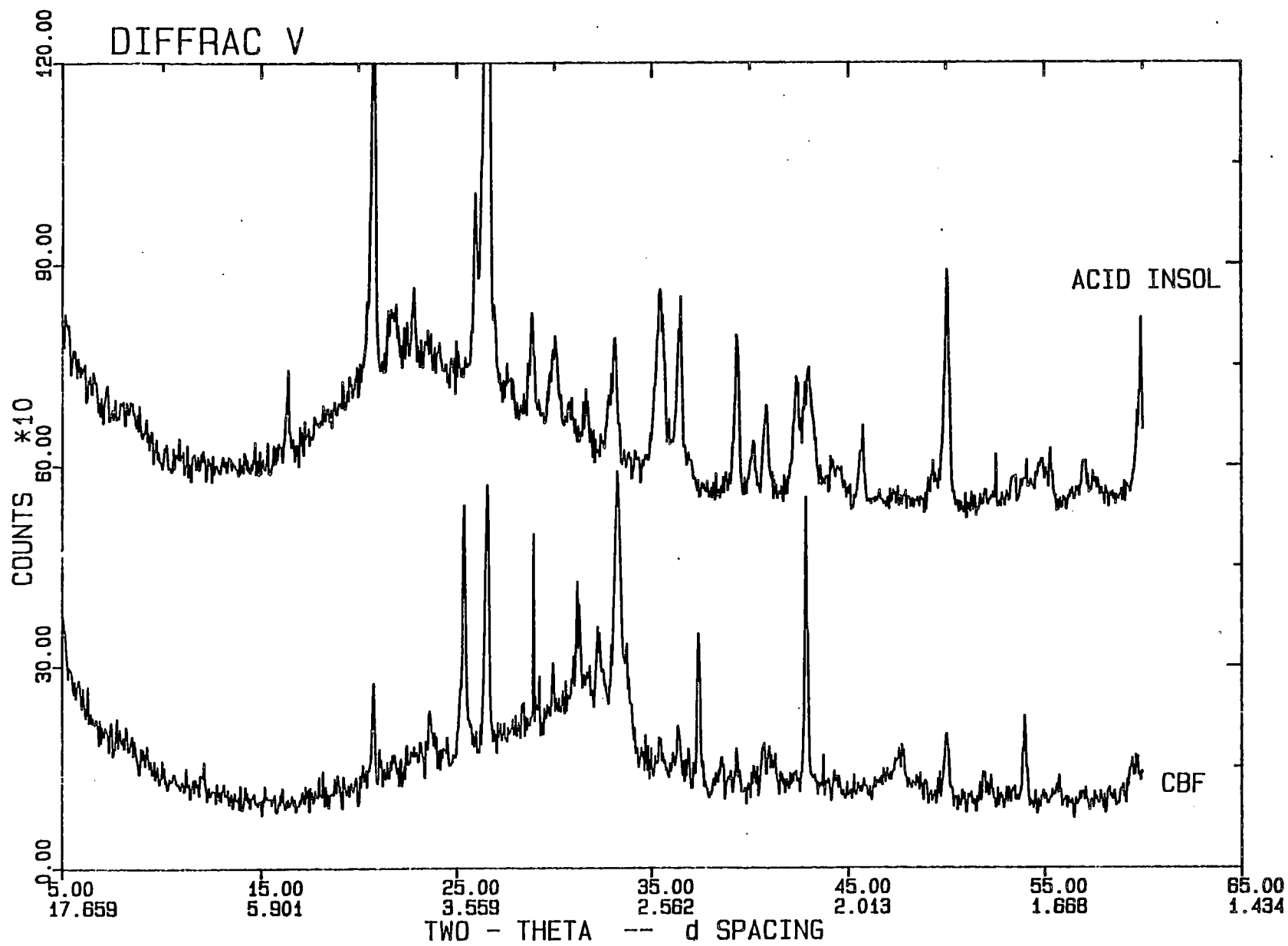


Figure 7. X-ray diffractogram of Council Bluffs fly ash.

Table II  
Compounds identified in the five fly ash samples

Compound	Clinton	Louisa	Ottumwa	Neal 4	Council Bluffs
Quartz, low ( $\text{SiO}_2$ , JCPDS #33-1161)	M	M	M	M	M
Anhydrite ( $\text{CaSO}_4$ , JCPDS #6-226)	m	M	M	M	M
Periclase ( $\text{MgO}$ , JCPDS# 4-829)	-	m	m	m	M
Lime ( $\text{CaO}$ , JCPDS# 4-777)	m	T	m	m	m
Ferrite Spinel [( $\text{Mg}$ , $\text{Fe}$ )( $\text{Fe}$ , $\text{Al}$ ) $_2\text{O}_4$ ]	M	m	m	m	m
Mullite ( $\text{Al}_6\text{Si}_2\text{O}_{13}$ , #15-776 )	M	m	m	m	m
Tricalcium Aluminate ( $\text{Ca}_3\text{Al}_2\text{O}_6$ , #38-1429)	-	M	M	M	M
Hematite ( $\text{Fe}_2\text{O}_3$ , JCPDS #33-664)	?	T	T	T	T
Tetracalcium Trialuminate Sulfate (JCPDS # 33-256)	-	-	m	-	m
Unknown	-	m	m	m	m

M= Major component    m = minor component    T = trace component    ? = question



All of the diffractograms (see Figures 3 through 7) indicated that a large amount of a given fly ash was amorphous to x-rays (i.e., glassy). Each of the class C fly ashes exhibited a glass scattering halo that reached a maximum intensity at about 30 degrees 2-theta (Cu K<sub>α</sub> radiation). The class F fly ash (Clinton) exhibited a glass scattering halo that reached a maximum intensity at about 23 degrees 2-theta.

To obtain additional information about the glass phases and minor components present in the five fly ash samples, the raw fly ashes were digested in hot acid (HCl) using a procedure described in an earlier IDOT report [5]. The acid insoluble residue was then subjected to X-ray analysis.

The results of XRD analysis has already been summarized in Table 2. Diffractograms of the acid insoluble residue from the five fly ash samples are shown in Figure 8. Please note that the acid insoluble residue from all the fly ashes contain very similar mineral assemblages. In fact, even the glassy material is quite similar (note how the glass scattering halo is constant at about 23 degrees 2-theta).

The results of XRF analysis on the acid insoluble fraction of the five fly ash samples is listed in Table III. In general, the XRF results are in excellent agreement with the XRD results, the acid insoluble residue is primarily composed of relatively inert siliceous material.

A study was conducted to assess the amount of alkalis (Na and K) that could be leached from the various samples of raw fly ash. This study should be applicable to the alkali durability portion of this research project. Briefly, the available alkali test procedure (see ASTM C 311-90 [4]) was used to extract and measure the amount of alkalis (expressed as equivalent Na<sub>2</sub>O) that were leached in to the solution after various curing times.

The preliminary results of this study are illustrated in Figure 9. Note in Figure 9, that the dissolution rate of fly ash alkalis is quite rapid for the first 14 to 28 days, then it decreases significantly. However, as is apparent in Figure 9, a considerable amount of alkalis are still being released into solution even at 9 weeks (63 days) of curing. Also, the Louisa and Neal 4 fly ashes, which have relatively low available alkali values at 28 days, either exceed or approach the ASTM C 618 available alkali specification limit (maximum = 1.50% equivalent Na<sub>2</sub>O) after 9 weeks of curing. This study is still continuing and additional information will be available in future reports.

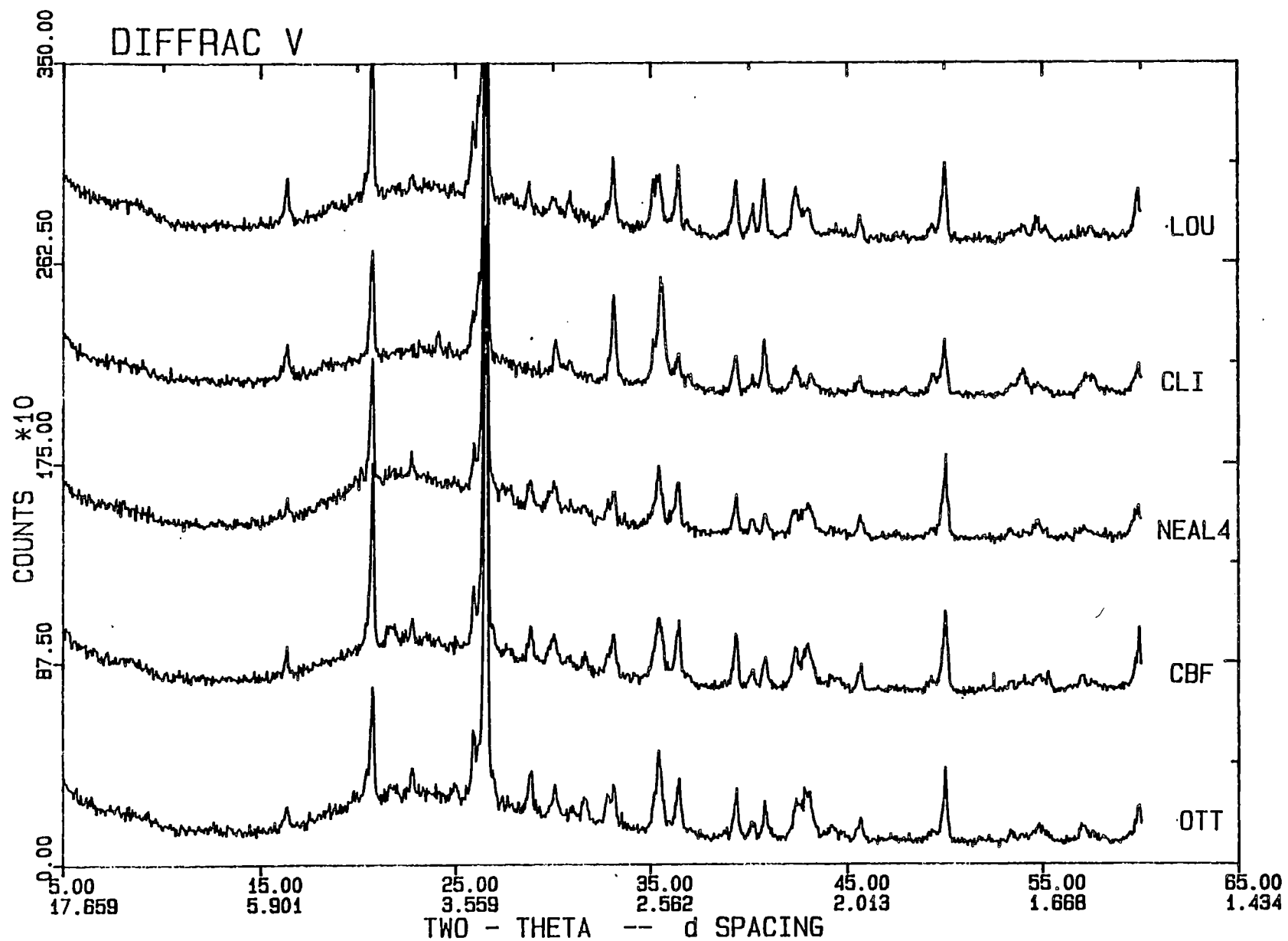


Figure 8. X-ray diffractograms comparing acid insoluble residue from the five sources of fly ash.

Table III

Summary of Chemistry of acid insoluble fraction of fly ashes

<u>Oxide (wt%)</u>	<u>Sample -----&gt;</u>				
	<u>Clinton</u>	<u>Louisa</u>	<u>Ottumwa</u>	<u>Neal 4</u>	<u>Council Bluffs</u>
SiO <sub>2</sub>	56.0	62.6	61.3	70.5	68.7
Al <sub>2</sub> O <sub>3</sub>	19.2	18.1	13.0	10.2	9.0
Fe <sub>2</sub> O <sub>3</sub> (T)	15.5	5.7	7.5	5.8	7.0
Sum:	90.7	86.3	81.8	86.5	84.7
SO <sub>3</sub>	0.0	0.3	0.9	0.5	0.7
CaO	0.7	3.9	4.9	4.4	4.7
MgO	0.9	1.3	1.4	1.4	1.8
P <sub>2</sub> O <sub>5</sub>	0.06	0.20	0.30	0.11	0.08
K <sub>2</sub> O	2.03	0.66	0.48	0.66	0.41
Na <sub>2</sub> O	0.2	0.8	1.9	1.5	2.2
TiO <sub>2</sub>	0.98	1.42	1.43	1.28	1.31
SrO	0.02	0.13	0.22	0.13	0.15
BaO	0.07	1.07	2.40	1.39	1.85
Loss on Ignition	0.98	1.75	1.89	1.92	1.21

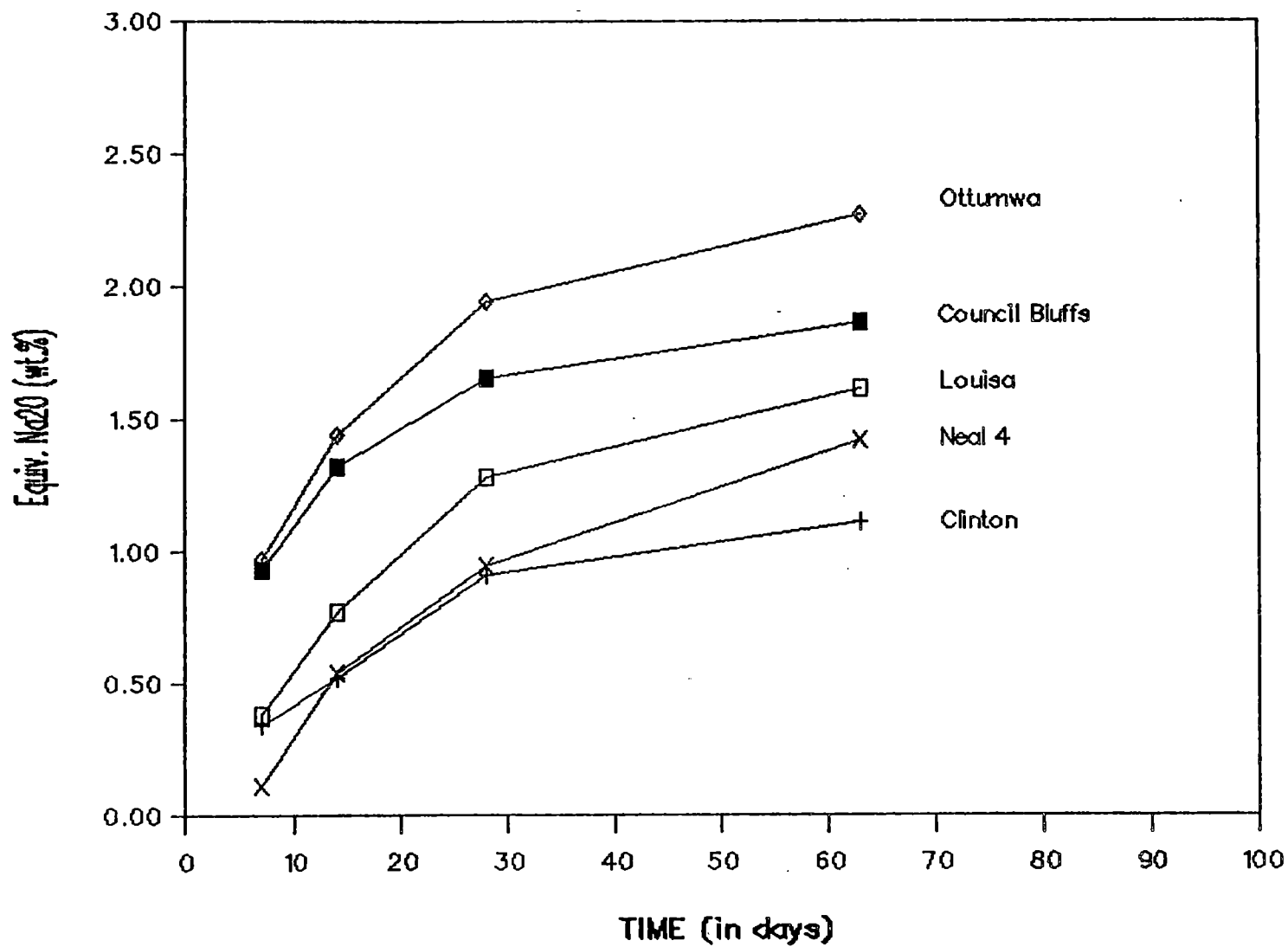


Figure 9. Preliminary results of alkali extraction tests on the five fly ashes used in this study.

### Portland Cements

The portland cements used in this study have been subjected to a series of physical and chemical tests. The cement tests were conducted in accordance with the ASTM methods specified for portland cements [3].

The chemical compositions of the three cements are summarized in Table IV. These assays were obtained by using the XRF techniques discussed earlier in this report. The amount of cement minerals present in each source of cement were calculated using the Bogue equations listed in ASTM C 150 [3]. Note that the Davenport cement had a chemical composition similar to a Type II cement; however, its  $C_3S + C_3A$  content was slightly high which placed it in the Type I portland cement category.

X-ray diffractograms of the three cements are shown in Figure 10. The results of the XRD tests were in good agreement with the XRF assays. The major compounds identified in the cements were alite (substituted tricalcium silicate; subst.- $C_3S$ ), belite (substituted dicalcium silicate; subst.- $C_2S$ ), a mineral close to tetracalcium aluminoferrite ( $C_4AF$ ) and tricalcium aluminate ( $C_3A$ ). Various sulfate bearing minerals were identified as minor constituents in the three cements. The Davenport cement contained bassanite, anhydrite and perhaps some gypsum; while the remaining two cements contained only gypsum and bassanite.

The physical properties of the three cements, namely normal consistency, compressive strength (C 109 mortar cubes), fineness and set time are listed in the lower portion of Table IV.

### Aggregates

X-ray diffractograms of the Jabens and Lamont aggregates (crushed stone for the concrete mixes) are shown in Figure 11. The Lamont stone is nearly a pure dolomite; however, it also contains a small amount of calcite and some quartz. The Jabens stone is basically a dolomitic limestone that contains a small amount of quartz. The results of XRF analysis are summarized in Table V.

The results of XRD and XRF analysis of the fine aggregates used in the alkali reactivity study are shown in Figure 12 and Table V, respectively. XRD analysis indicated that the pyrex glass was amorphous to the x-rays, the ASTM C 109 sand was nearly pure quartz and that the Class V aggregate was a mixture of quartz and feldspar minerals.

Table IV  
Chemical and Physical Characteristics of the portland cements

Chemcial Properties:	Sample		
<u>Oxide (wt%)</u>	<u>South Dakota</u>	<u>Dundee</u>	<u>Davenport</u>
SiO <sub>2</sub>	22.57	21.27	21.03
Al <sub>2</sub> O <sub>3</sub>	3.53	4.70	4.23
Fe <sub>2</sub> O <sub>3</sub>	3.43	1.93	2.80
CaO	64.6	64.4	63.0
MgO	1.06	2.11	3.43
SO <sub>3</sub>	1.94	3.08	2.63
Loss on Ignition	1.07	1.16	1.04
Na <sub>2</sub> O	0.10	0.05	0.15
K <sub>2</sub> O	0.33	0.43	1.0
TiO <sub>2</sub>	0.16	0.17	0.24
P <sub>2</sub> O <sub>5</sub>	0.08	0.01	0.12
SrO	0.13	0.03	0.06
Calculated Cement Compounds (wt%)			
C <sub>3</sub> S	55.7	56.2	54.3
C <sub>2</sub> S	22.7	18.6	19.3
C <sub>3</sub> A	4.2	9.7	7.4
C <sub>4</sub> AF	10.4	5.9	8.5
CEMENT TYPE	V	I	I (close to II)
Physical properties:			
Normal consistency(%)	24.8*	25.0*	24.8*
Compressive strength (psi)			
@ 1 day	1820	2120	1980
@ 2 days	2940	3090	2780
@ 3 days	3640	3660	3110
Blaine Fineness(m <sup>2</sup> /Kg)	378*	403*	386*
Vicat Initial Set Time	OK*	OK*	OK*

\* = value from IDOT test report

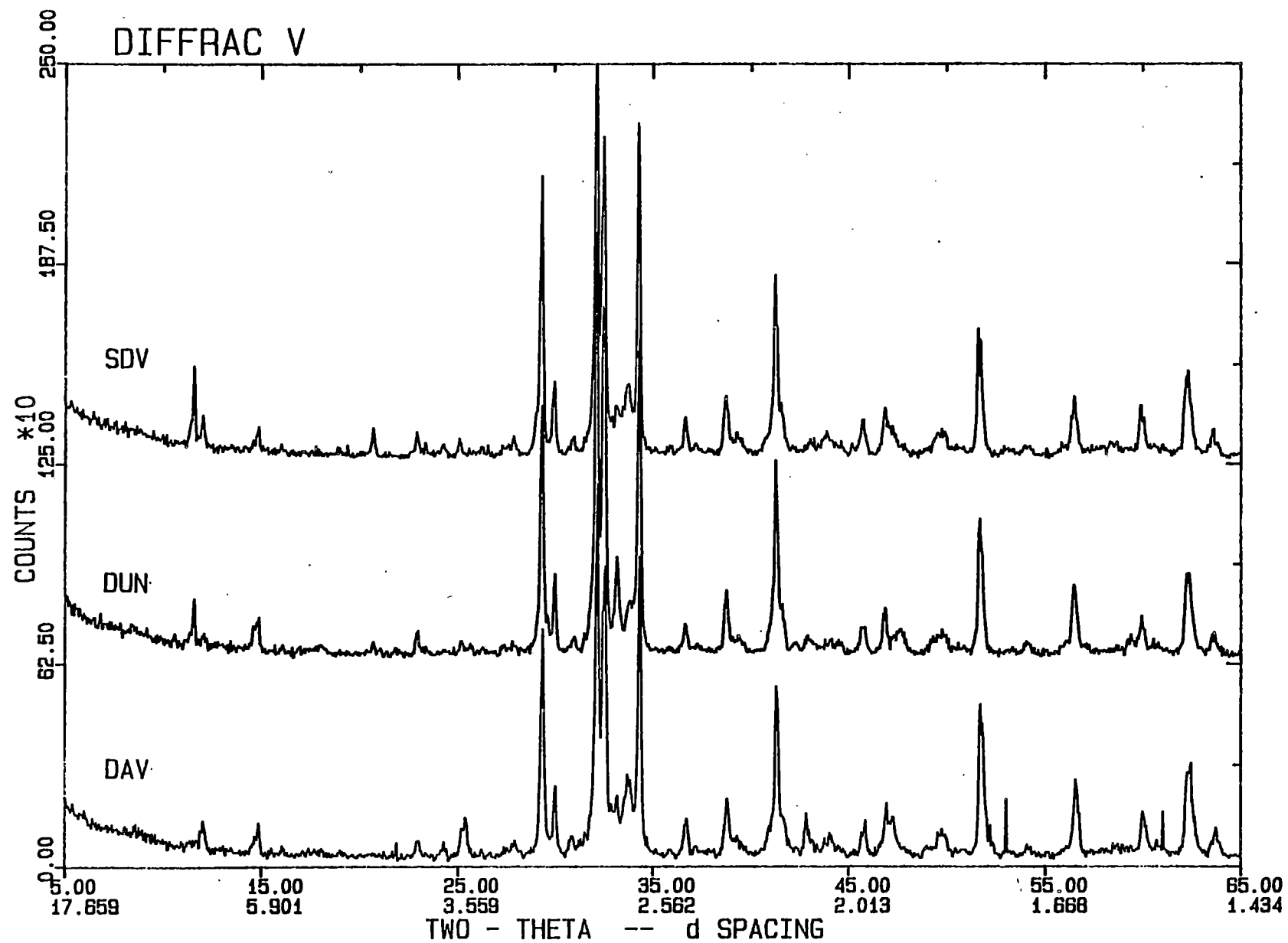


Figure 10. X-ray diffractograms of the three cements used in this study.

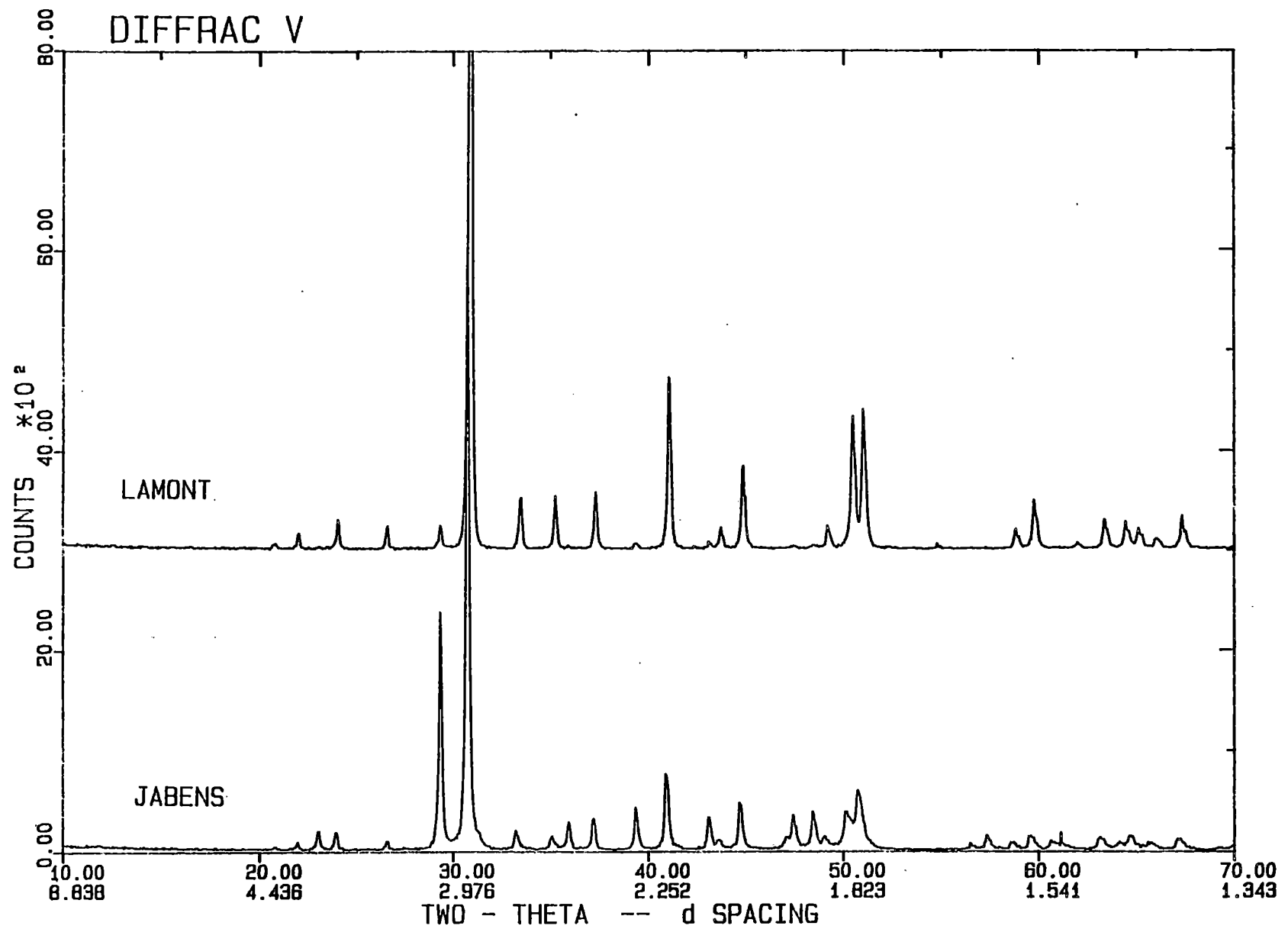


Figure 11. X-ray diffractograms of the two coarse aggregate sources used in the concrete portion of this study.



Table V  
Results of XRF analysis of the aggregate samples

<u>Oxide (wt%)</u>	<u>Jabens</u>	<u>Lamont</u>	<u>Pyrex Glass*</u>	<u>Class V</u>	<u>C109 Sand</u>
SiO <sub>2</sub>	1.61	1.82	85.1	81.7	98.5
Al <sub>2</sub> O <sub>3</sub>	0.21	0.20	3.0	10.2	0.1
Fe <sub>2</sub> O <sub>3</sub>	0.65	0.22	0.2	1.1	0.7
CaO	35.2	30.5	0.02	0.5	0.1
MgO	14.7	20.9	N/M	1.1	0.1
SO <sub>3</sub>	0.38	0.02	<0.1	<0.1	<0.1
K <sub>2</sub> O	0.08	0.07	0.02	3.2	<0.01
TiO <sub>2</sub>	0.02	0.02	N/M	N/M	N/M
P <sub>2</sub> O <sub>5</sub>	0.02	<0.01	N/M	N/M	N/M
SrO	0.02	0.02	N/M	N/M	N/M
MnO	0.04	0.01	N/M	N/M	N/M
Na <sub>2</sub> O	N/M	N/M	4.9	2.1	<0.1
Loss on Ignition	44.5	46.0	0.3	0.25	0.04

-----  
N/M - not measured

\* - average composition of two lots of pyrex glass

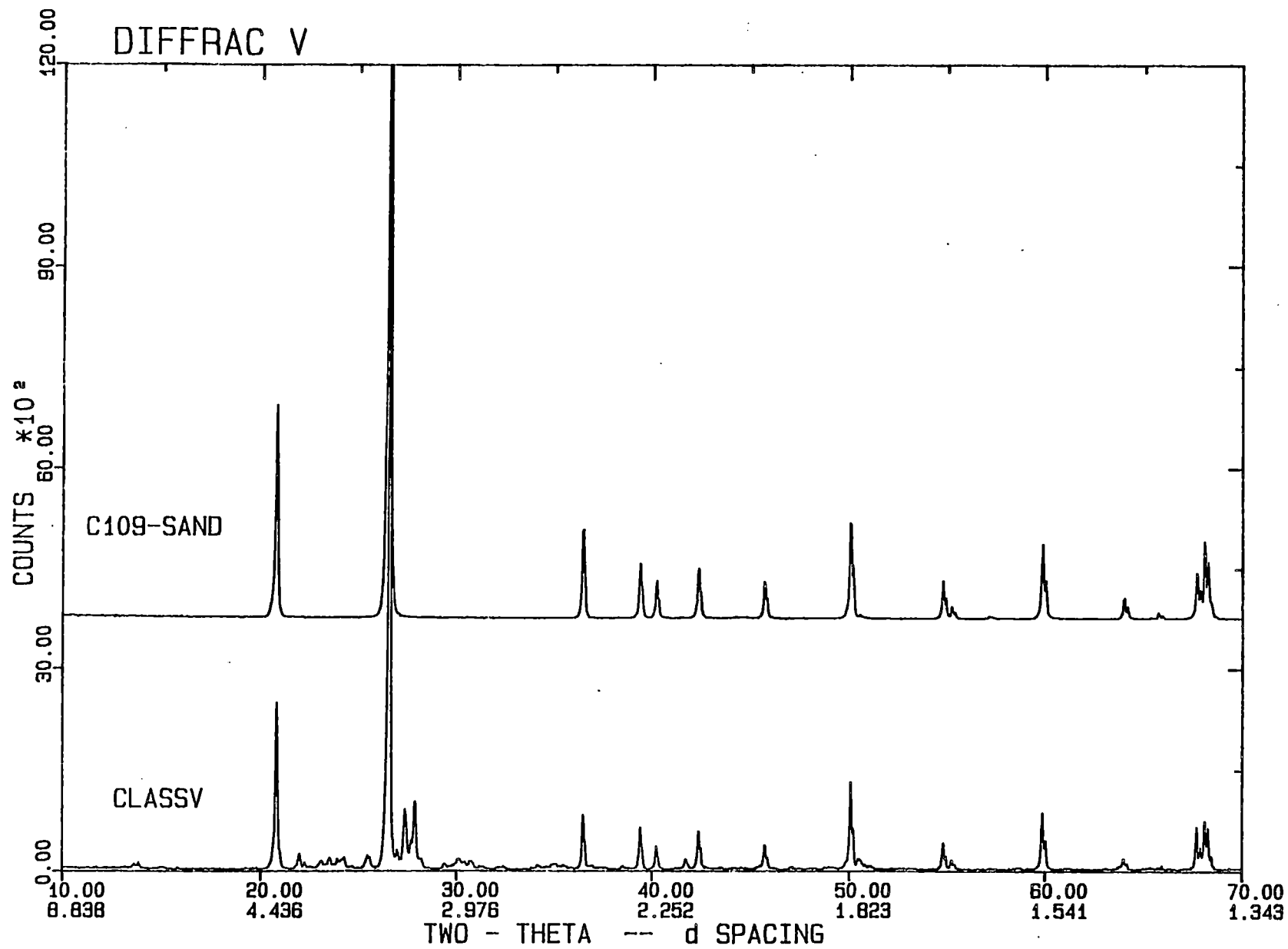


Figure 12. X-ray diffractograms of two of the fine aggregates used in the alkali durability portion of this study.

### Reagent and Technical Grade Materials

The results of XRD analysis of the sodium chloride and sodium sulfate used during the first year of this project is shown in Figures 13 and 14, respectively. The major compounds in the various diffractograms correspond to the desired material (i.e., sodium chloride or sodium sulfate). The results of XRF analysis are summarized in Table VI. In general, the technical grade materials compare very well with the reagent grade materials.

### Sulfate Durability Tests

The results of the portland cement fly ash paste testing portion of this research project will not be presented in this report. The tests are currently underway but they were only initiated in the past few months; and hence, only a few data points are currently available for each fly ash-cement paste specimen.

The results of the mortar bar study are shown in Figures 15 through 30. The vertical axis on each figure represents specimen growth (% expansion) while the horizontal axis depicts time (in days since the sample was submerged in the sulfate bearing solution). Each figure consists of two separate graphs. The upper graph depicts the results of the standard (ASTM C 1012) tests which used a sulfate concentration of 5% (wt %  $\text{Na}_2\text{SO}_4$ ). The lower graph depicts the results of the tests that used the mixed salt treatment (i.e., 9.5% NaCl, 0.25%  $\text{Na}_2\text{SO}_4$ ). Note that there is a difference in the vertical scale used on the upper and lower graph in each figure, this was done to magnify the expansions observed in the mixed salt treatment.

The following abbreviations are used throughout the various figures:

- DUN - Dundee cement
- DAV - Davenport cement
- SDV - South Dakota cement
- CLI - Clinton fly ash
- LOU - Louisa fly ash
- CBF - Council Bluffs fly ash
- NE4 - Neal 4 fly ash
- OTT - Ottumwa fly ash

Please remember that fly ash replacements of 7.5, 15, 22.5 and 30% (by weight for an equivalent weight of cement) were utilized in this mortar study. The fly ash replacements are listed in the legend of each figure.

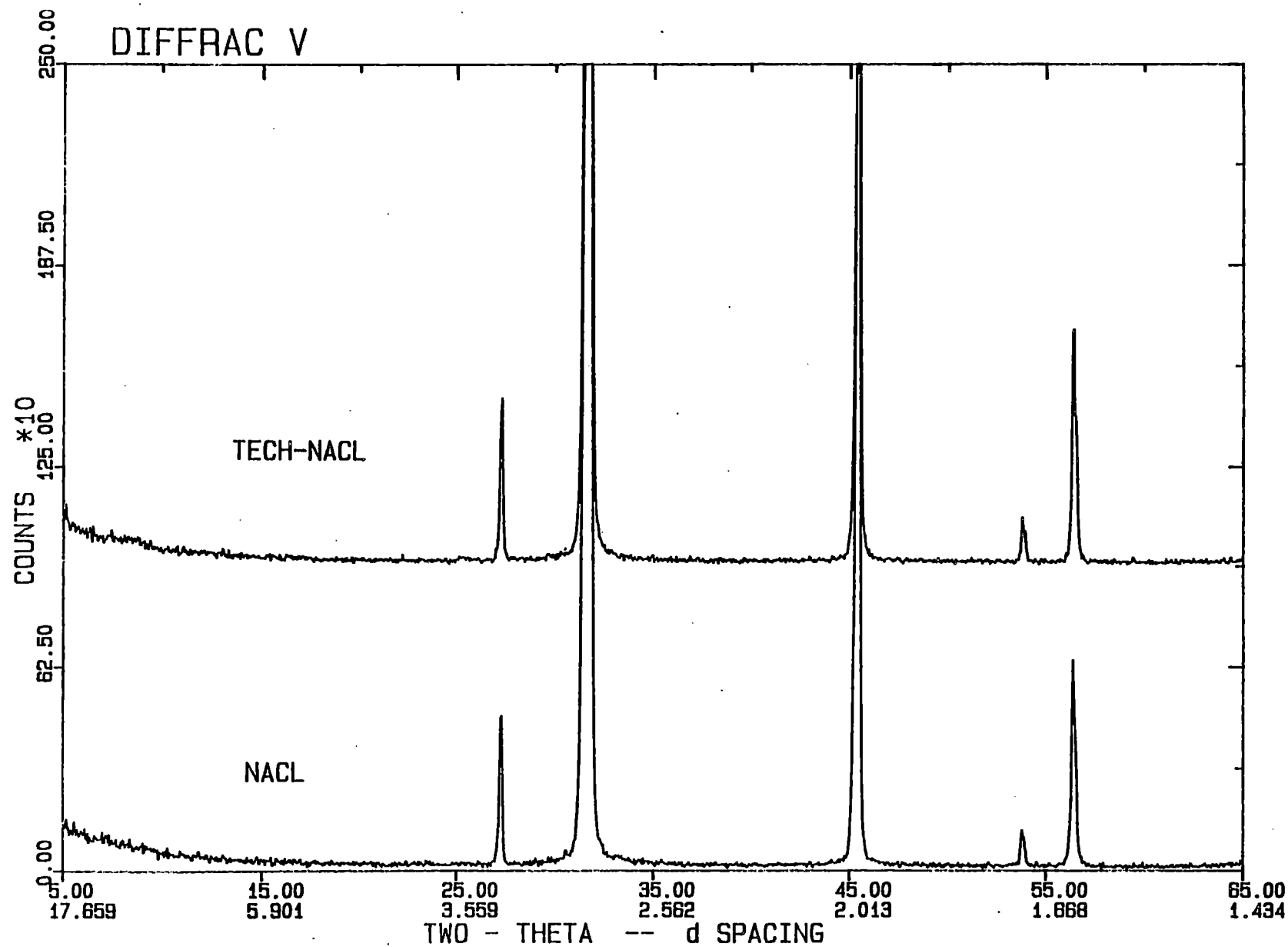


Figure 13. X-ray diffractograms of the reagent and technical grade sodium chloride used in this study.

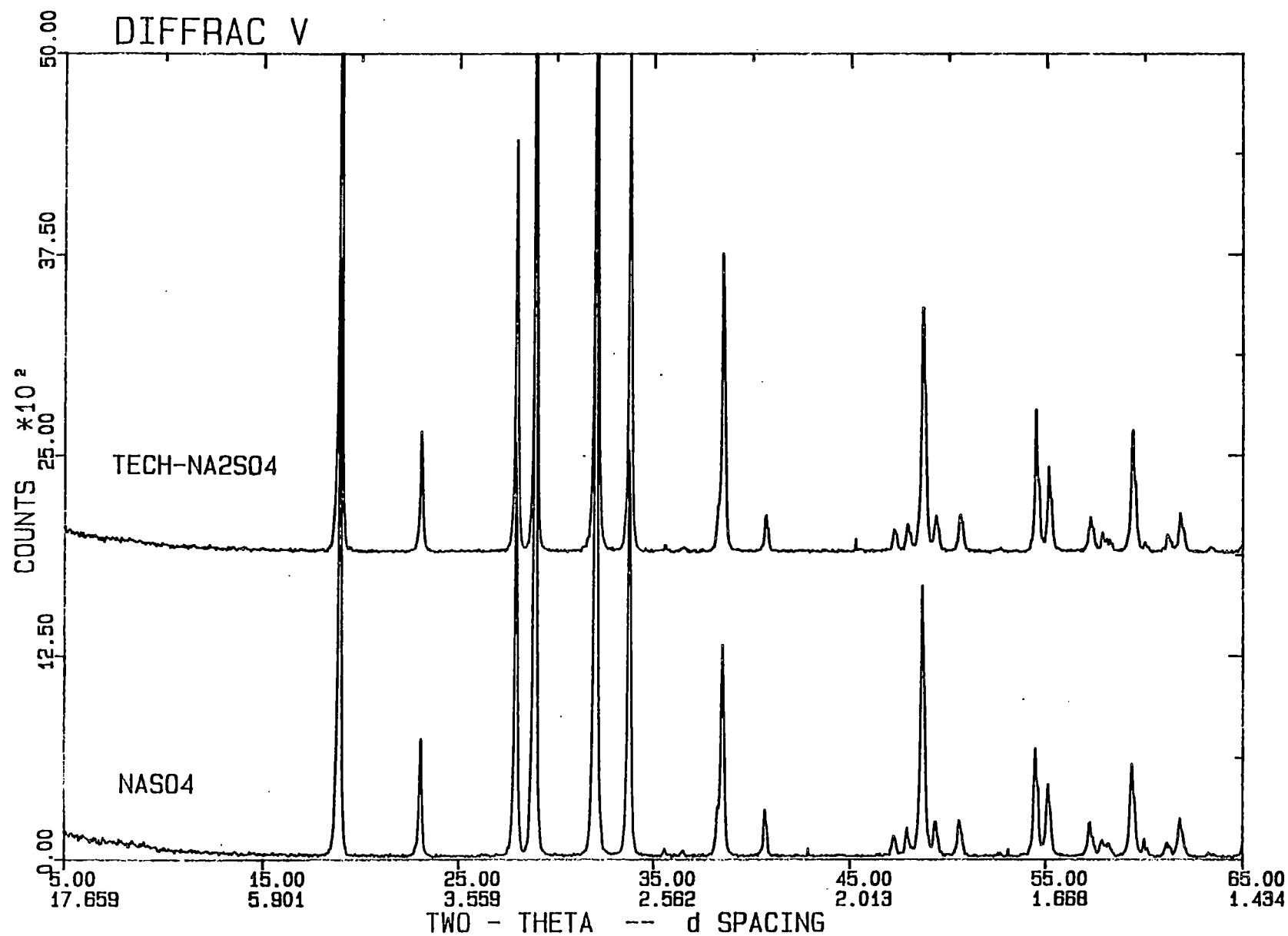


Figure 14. X-ray diffractograms of the reagent and technical grade sodium sulfate used in this study.

Table VI

Results of XRF analysis on Reagent and Technical grade chemicals  
(all values in parts per million (ppm) unless noted otherwise)

element	Sample			
	Reagent Grade		Technical Grade (Lot #1)	
	NaCl	Na <sub>2</sub> SO <sub>4</sub>	NaCl	Na <sub>2</sub> SO <sub>4</sub>
Sr	9	14	16	18
Br	61	<10	100	<10
Mg	0.02%	<50	70	0.02%
Fe	50	50	50	60
Ca	<10	85	0.17%	30
K	30	20	0.01%	0.02%
Ti	17	12	17	16
Ba	<10	<10	<10	<10
Cl	N/M	40	N/M	0.22%
I	<10	<10	<10	<10
Si	<50	<50	<50	<50
S	70	N/M	0.12%	N/M
P	<10	<10	<10	<10
Al	65	60	<50	<50

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N/M = not measured

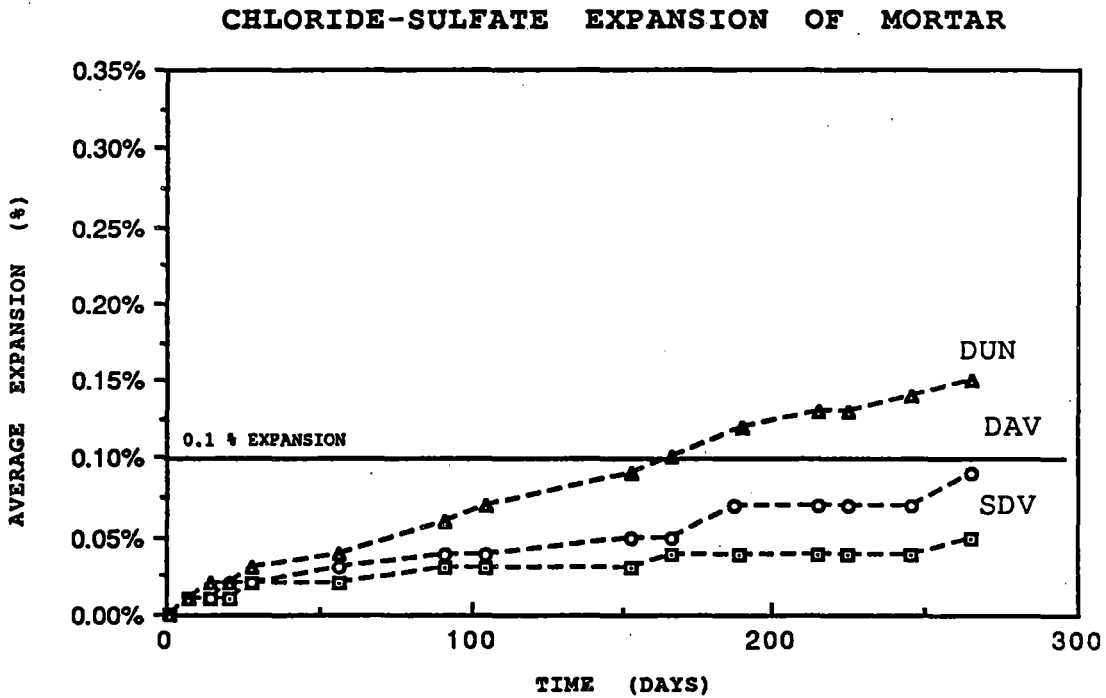
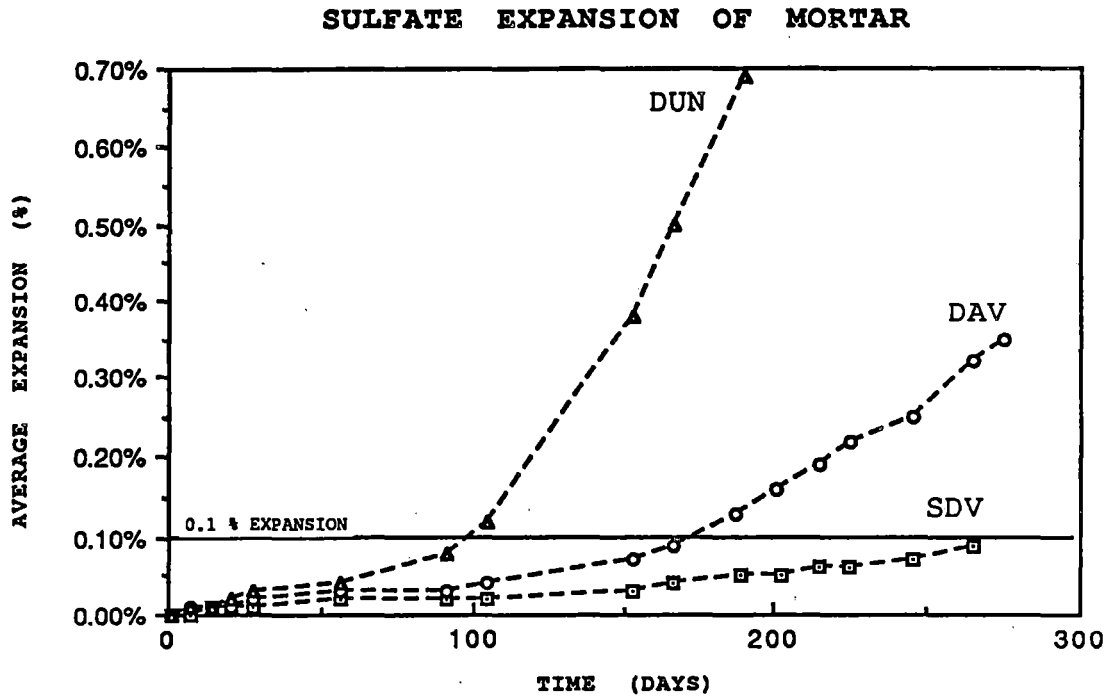


Figure 15. Average expansion versus time for mortar specimens containing the three cements used in this study.

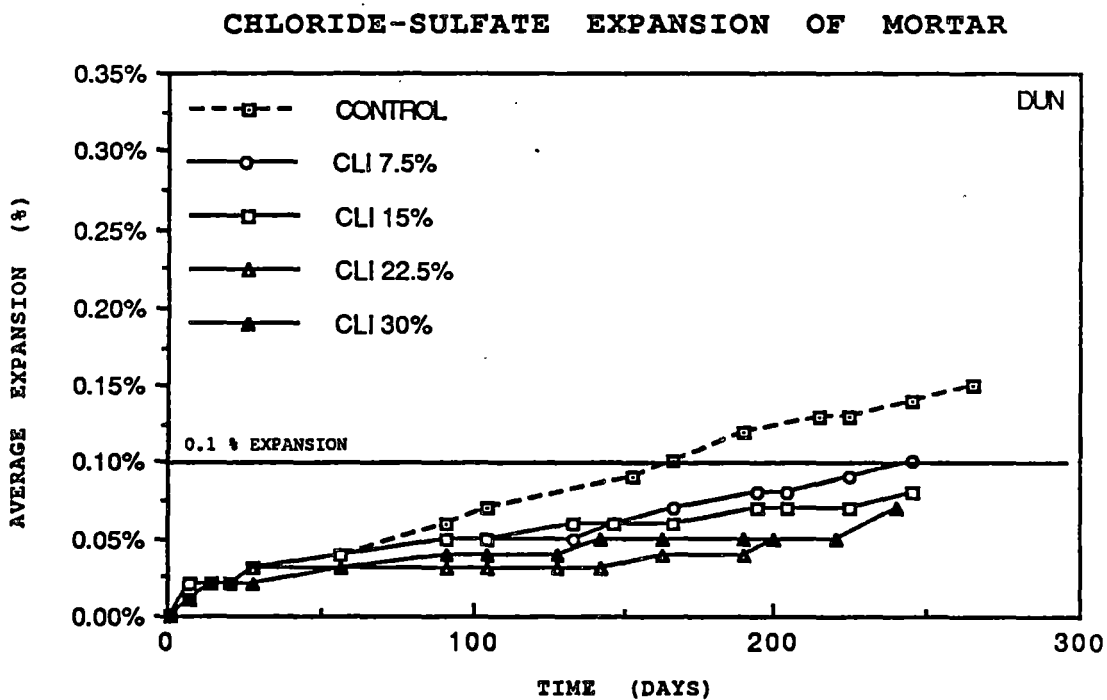
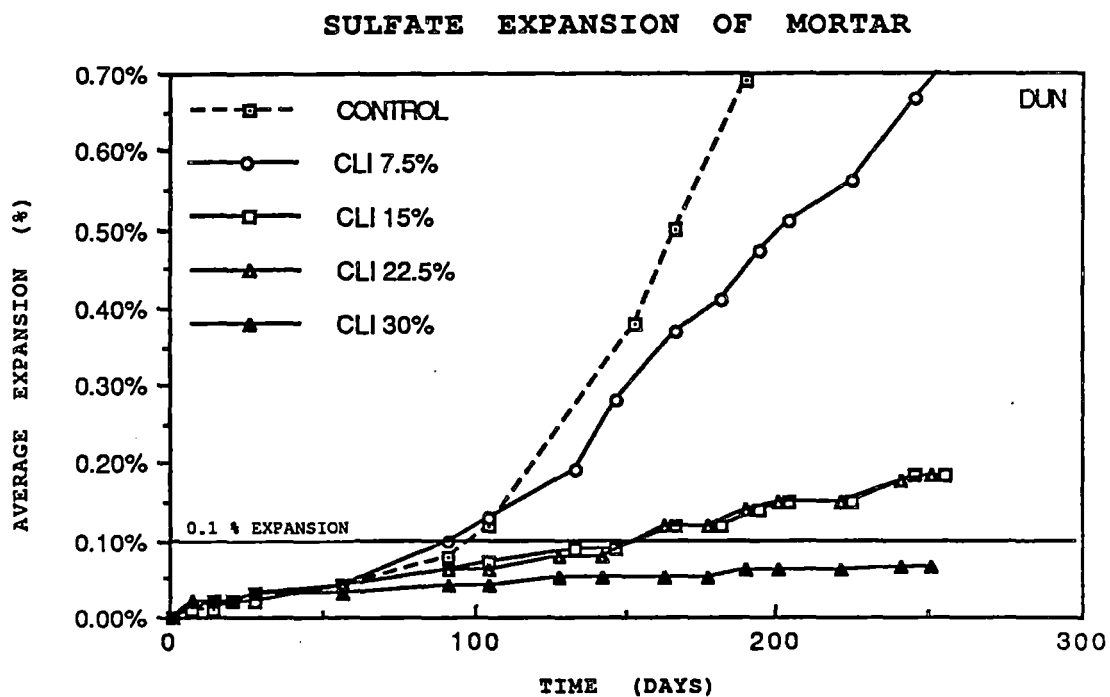


Figure 16. Average expansion versus time for mortar specimens containing Clinton fly ash and Dundee cement.



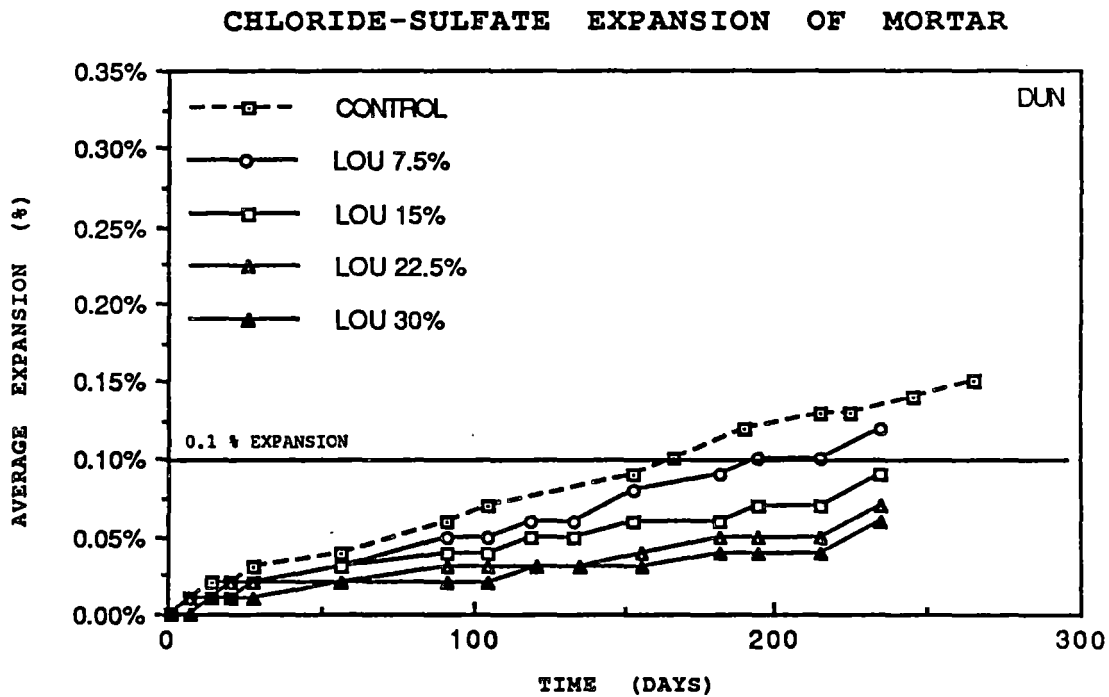
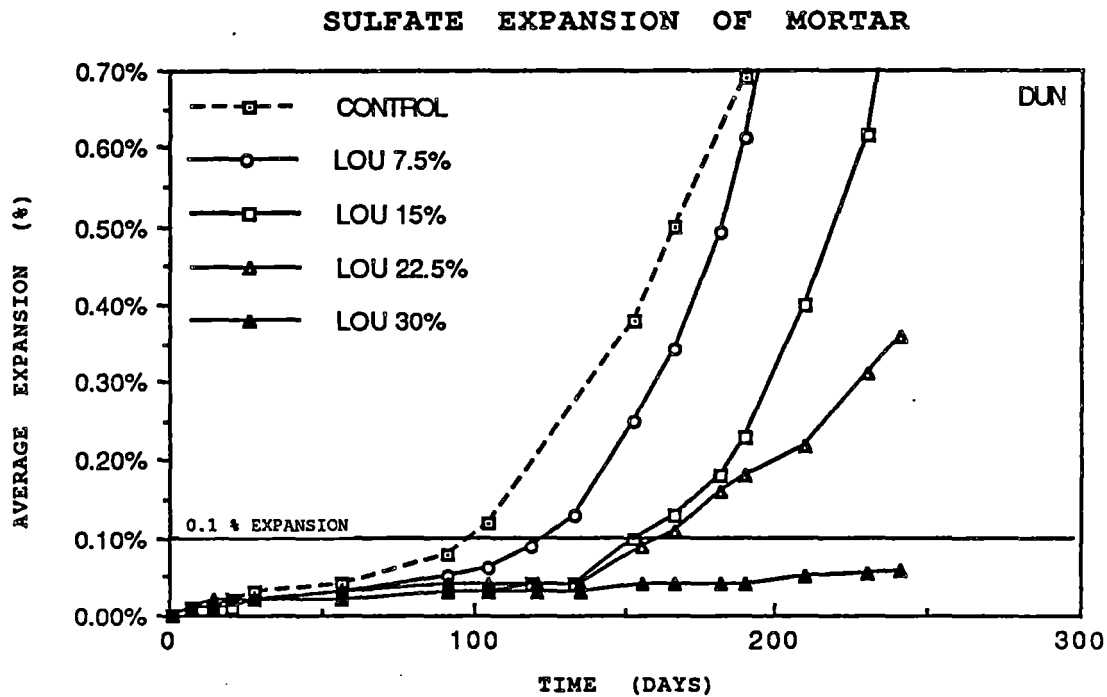


Figure 17. Average expansion versus time for mortar specimens containing Louisa fly ash and Dundee cement.

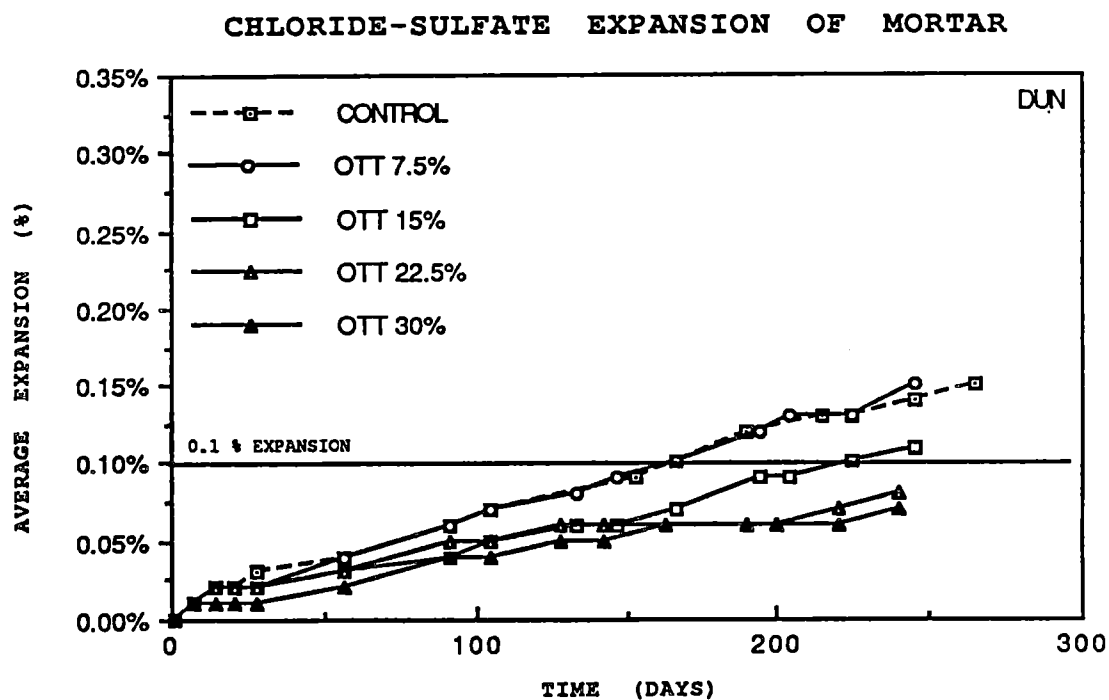
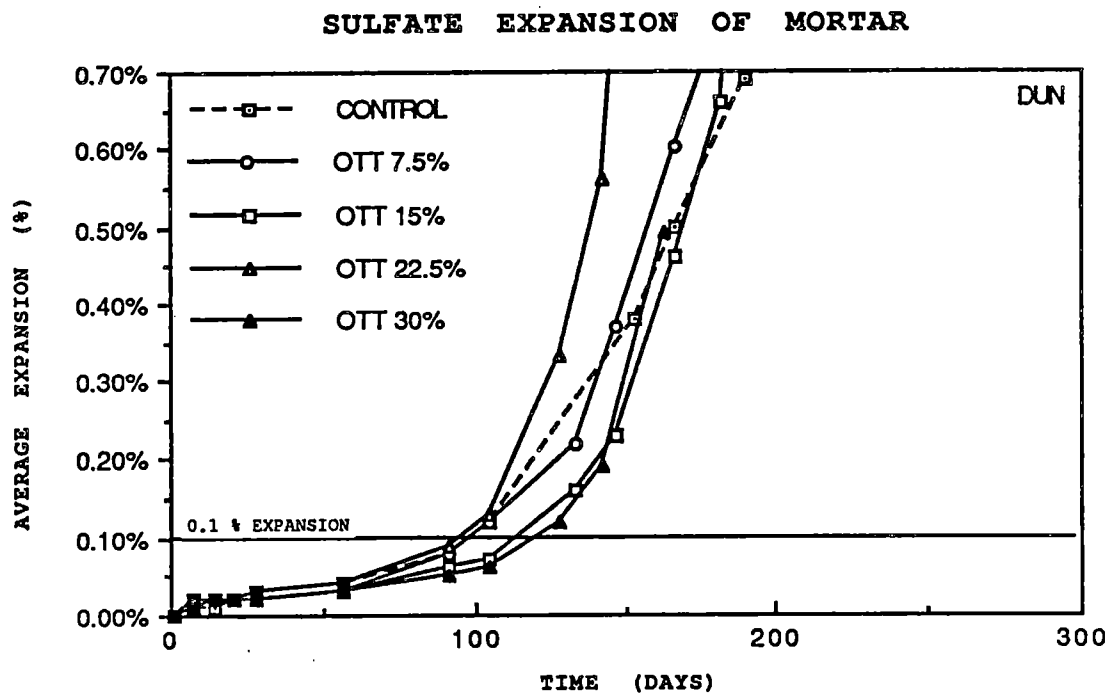


Figure 18. Average expansion versus time for mortar specimens containing Ottumwa fly ash and Dundee cement.

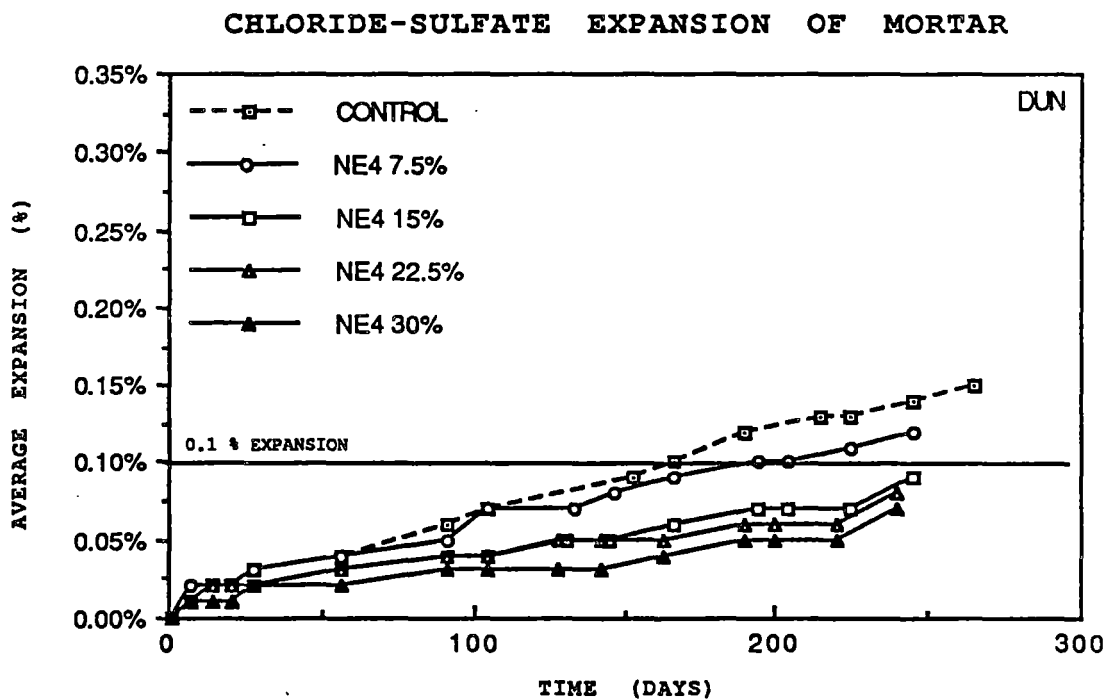
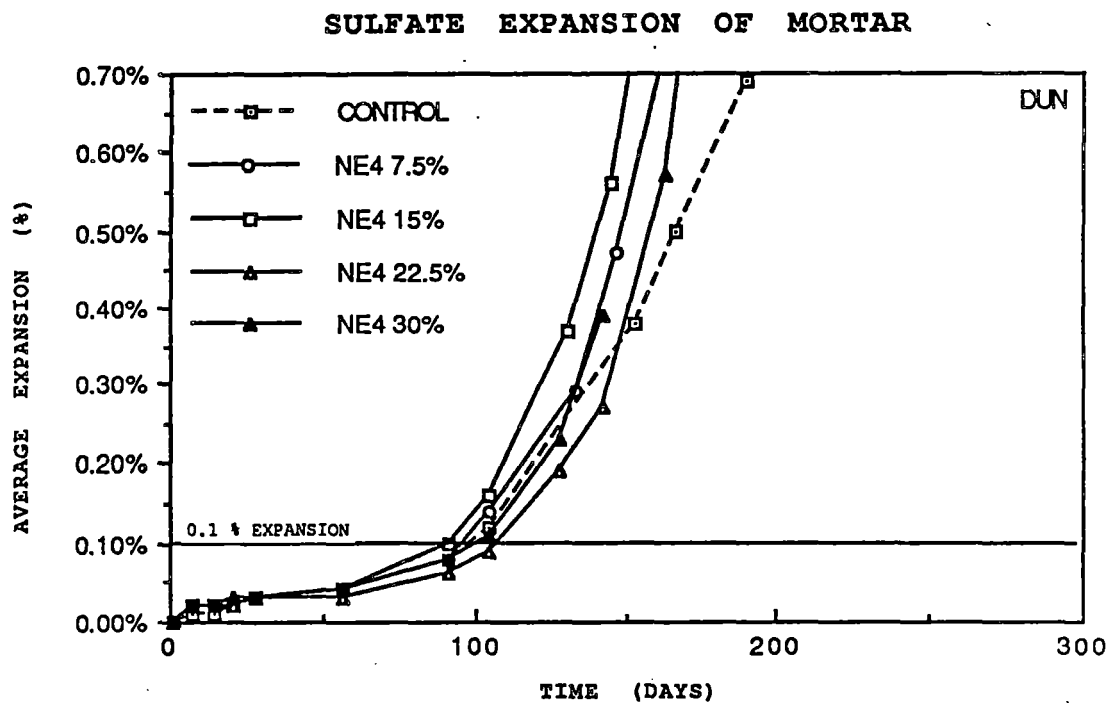


Figure 19. Average expansion versus time for mortar specimens containing Neal 4 fly ash and Dundee cement.

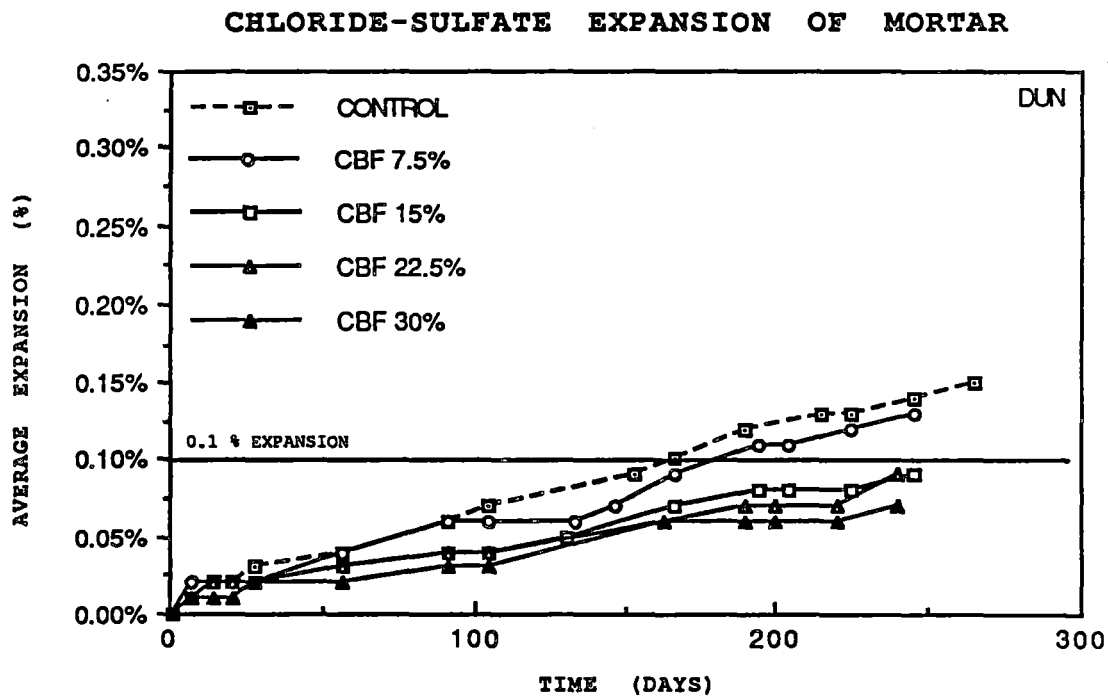
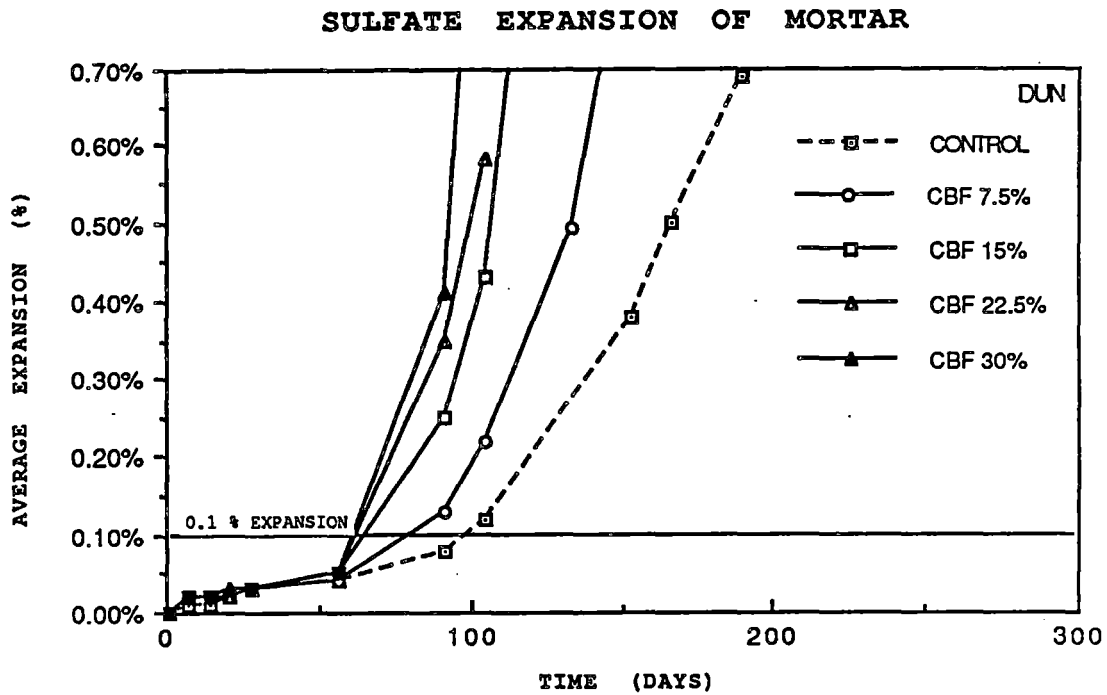


Figure 20. Average expansion versus time for mortar specimens containing Council Bluffs fly ash and Dundee cement.

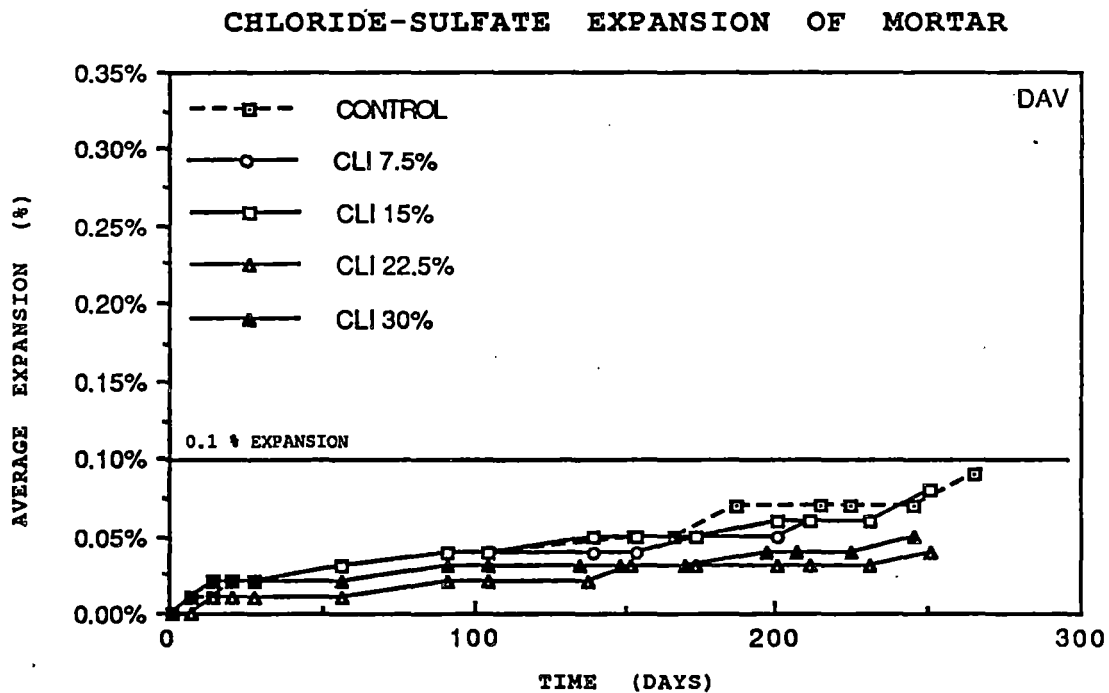
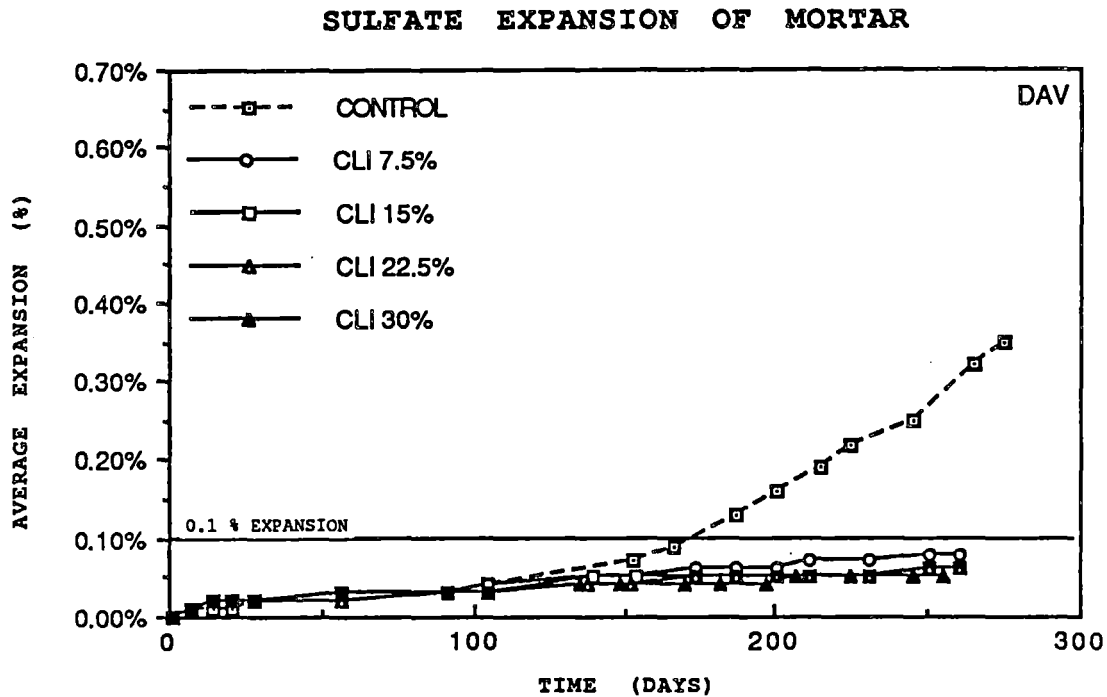


Figure 21. Average expansion versus time for mortar specimens containing Clinton fly ash and Davenport cement.

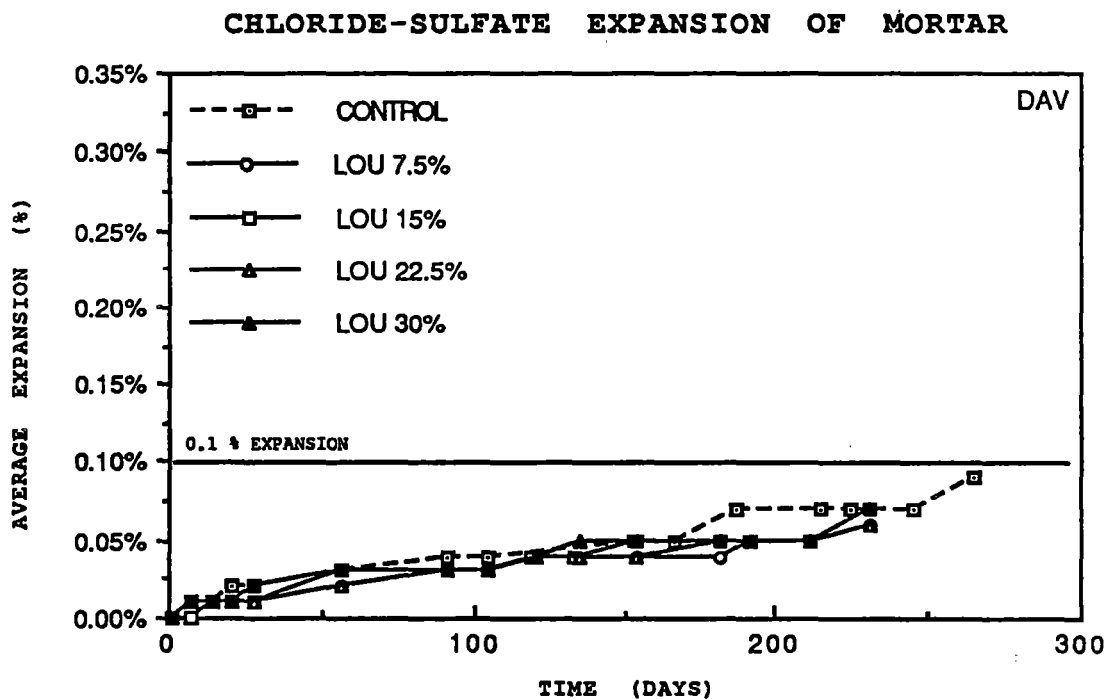
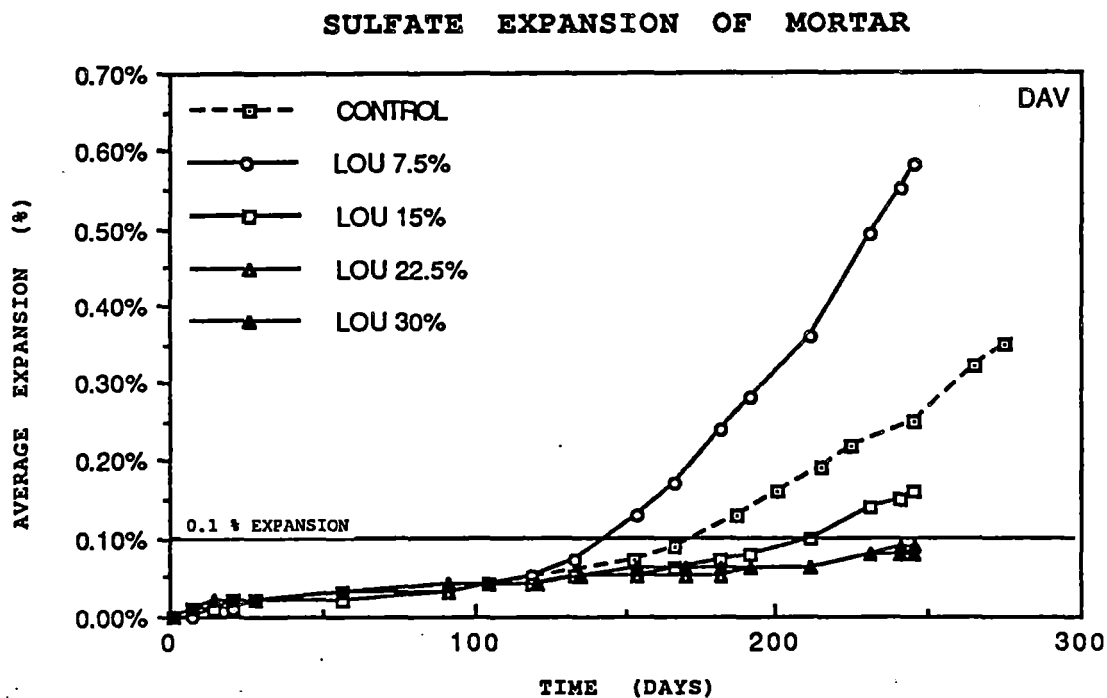


Figure 22. Average expansion versus time for mortar specimens containing Louisa fly ash and Davenport cement.

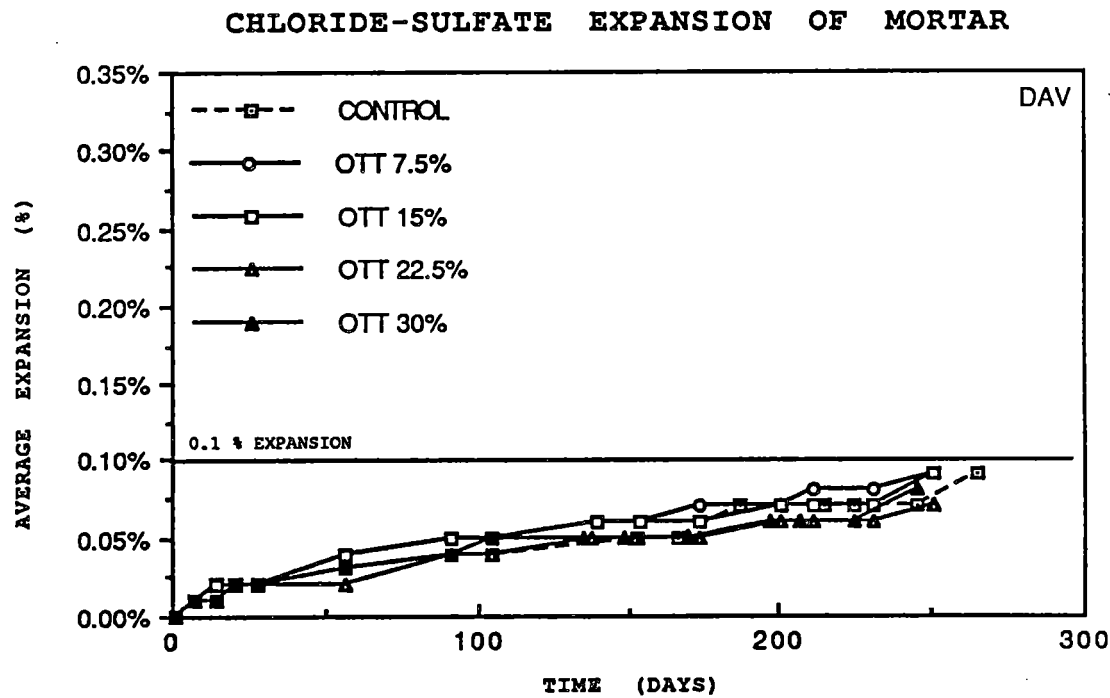
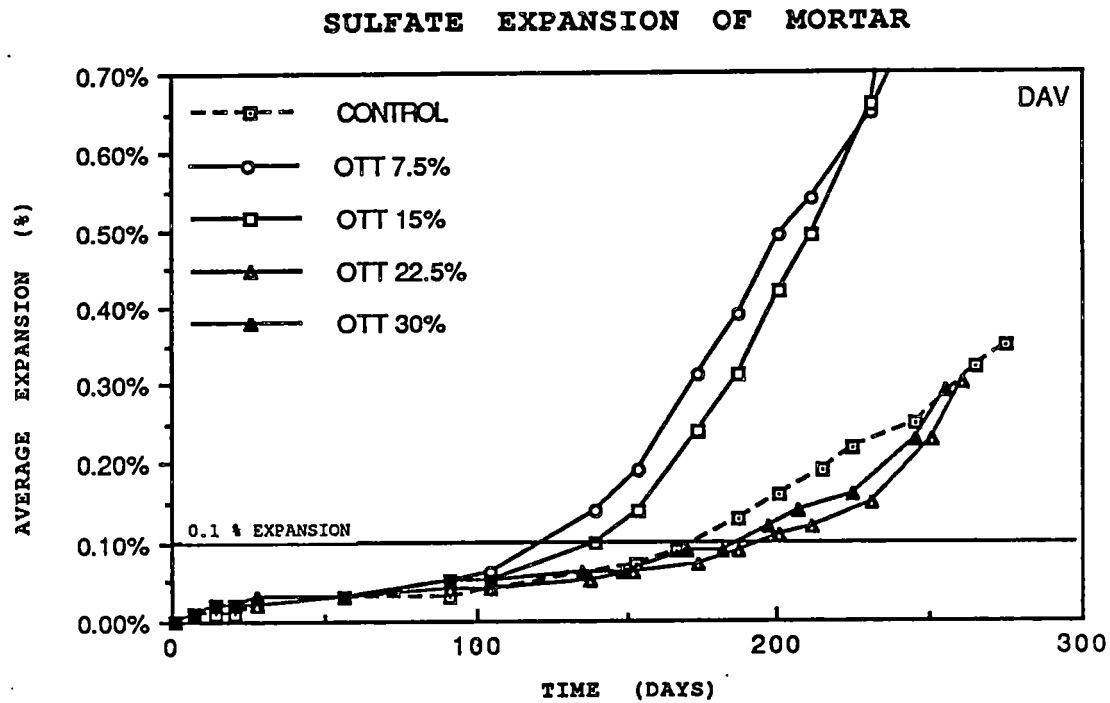


Figure 23. Average expansion versus time for mortar specimens containing Ottumwa fly ash and Davenport cement.

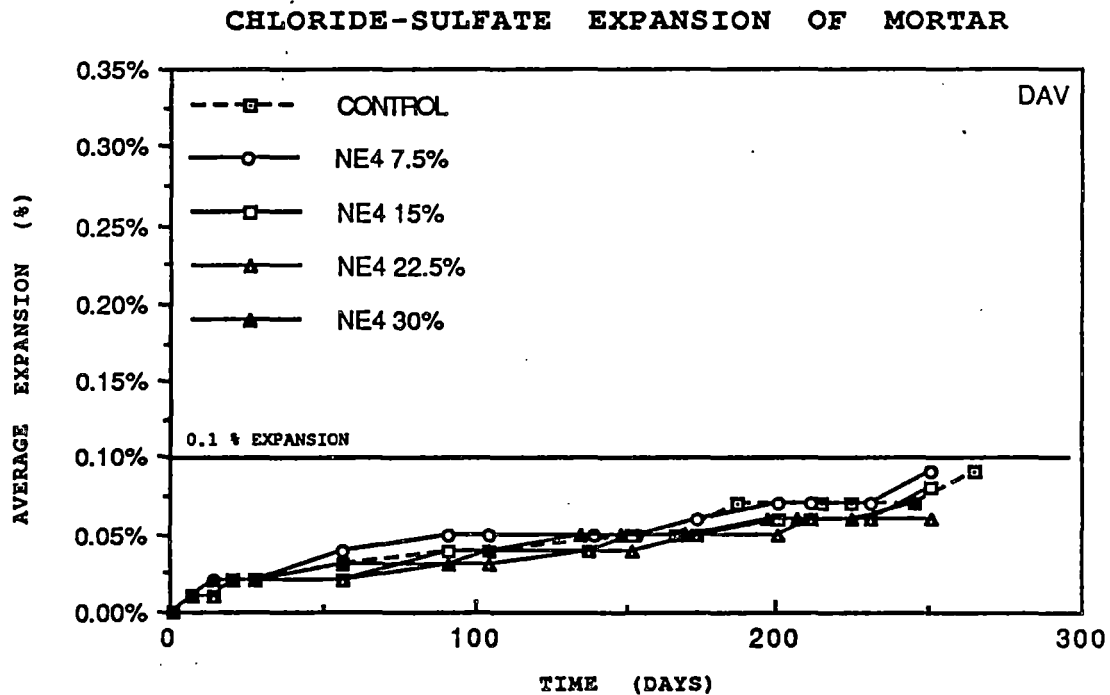
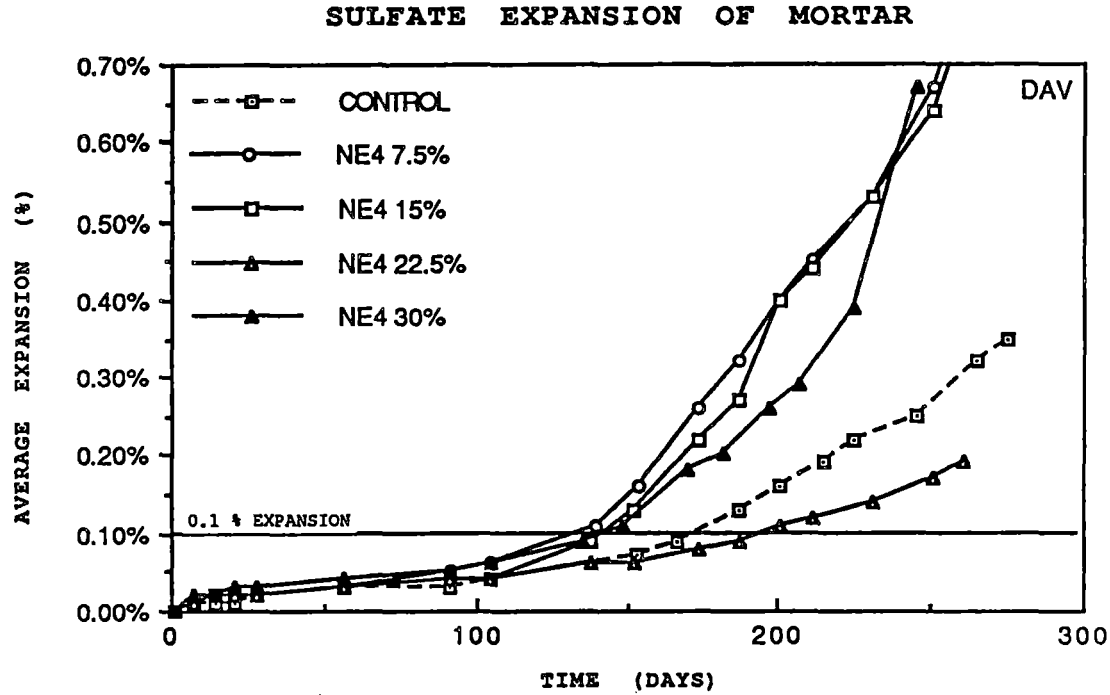


Figure 24. Average expansion versus time for mortar specimens containing Neal 4 fly ash and Davenport cement.



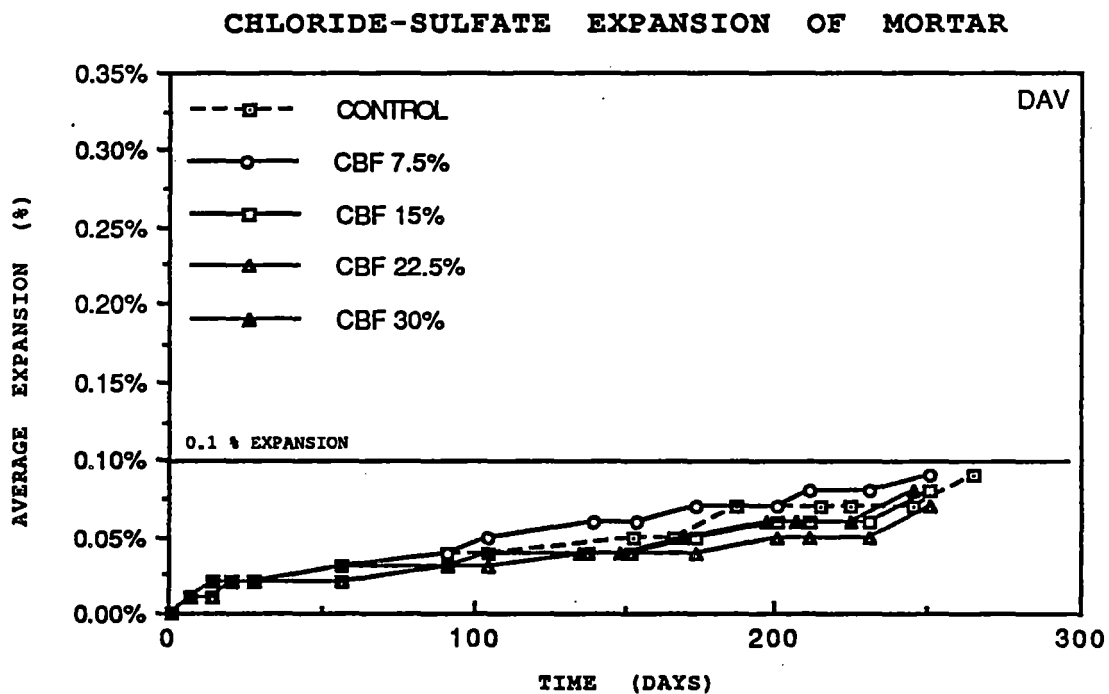
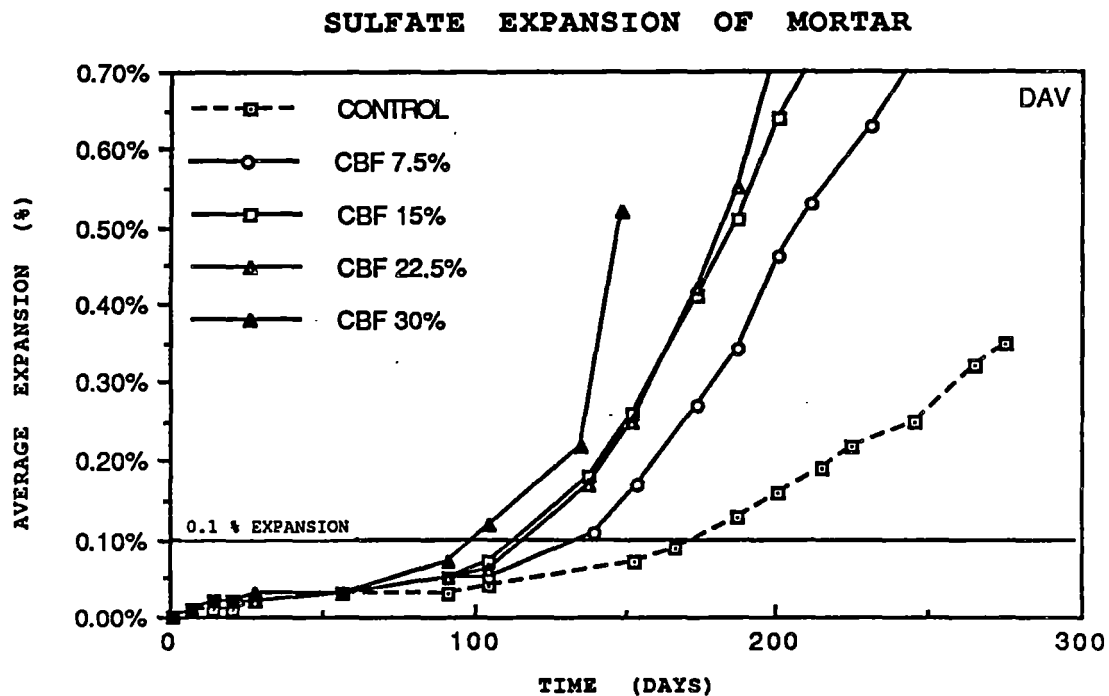
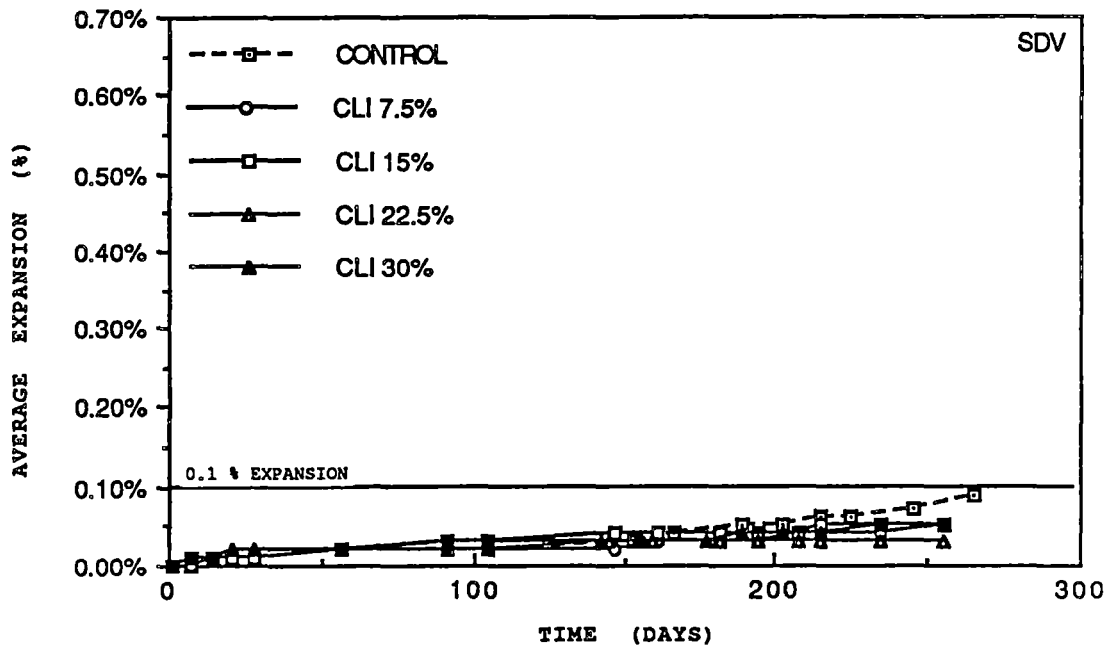


Figure 25. Average expansion versus time for mortar specimens containing Council Bluffs fly ash and Davenport cement.

### SULFATE EXPANSION OF MORTAR



### CHLORIDE-SULFATE EXPANSION OF MORTAR

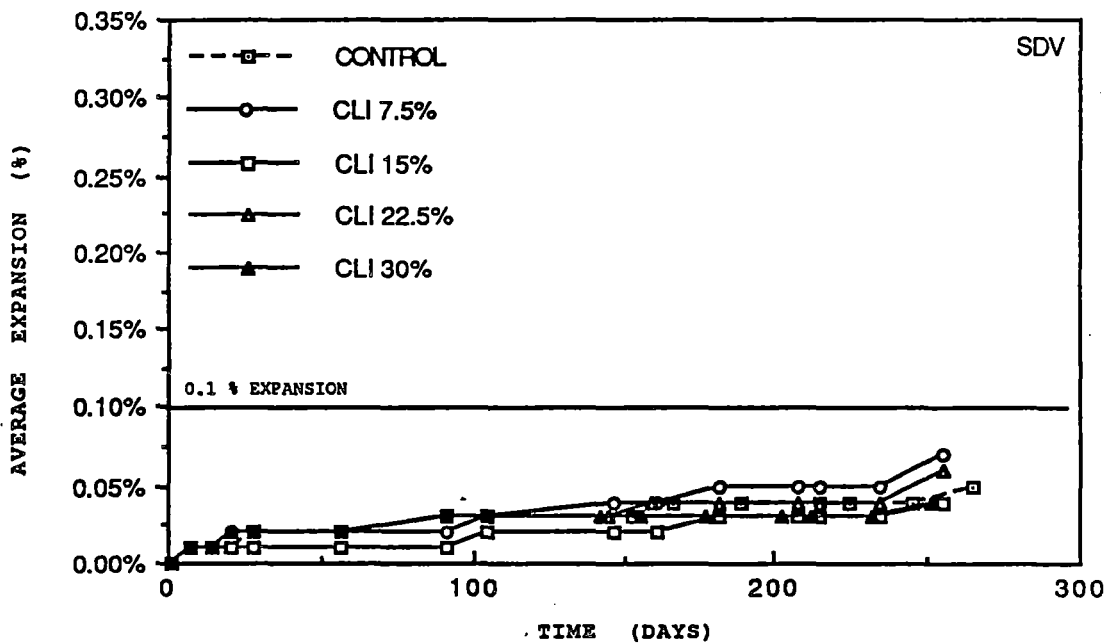


Figure 26. Average expansion versus time for mortar specimens containing Clinton fly ash and South Dakota cement.

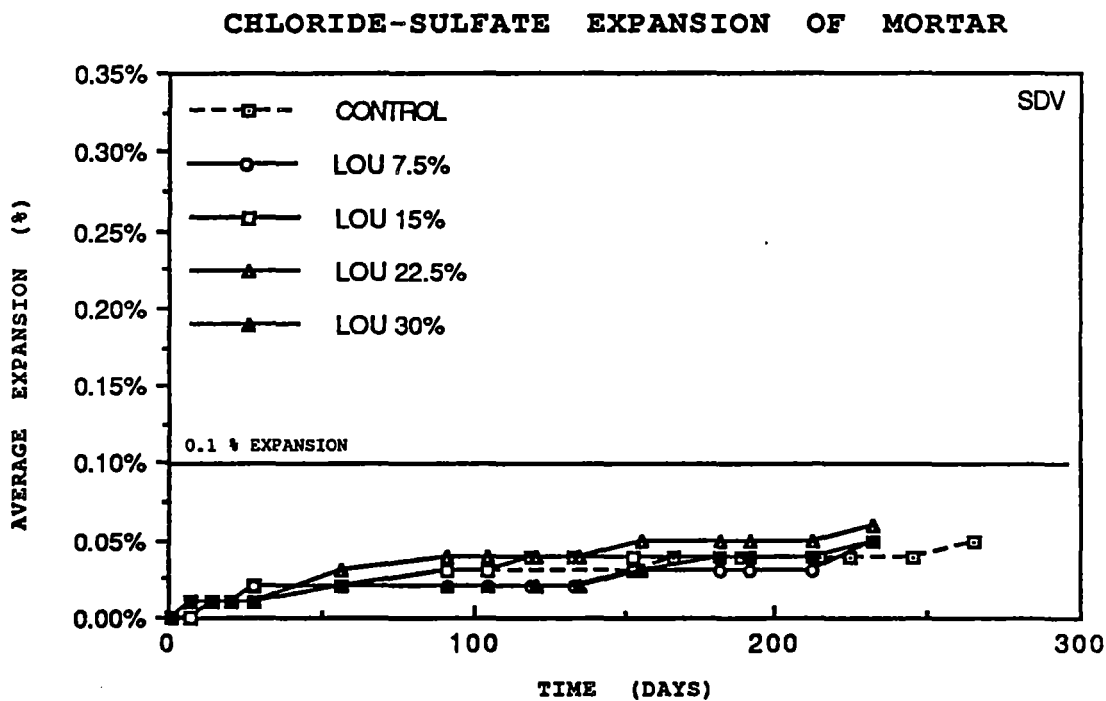
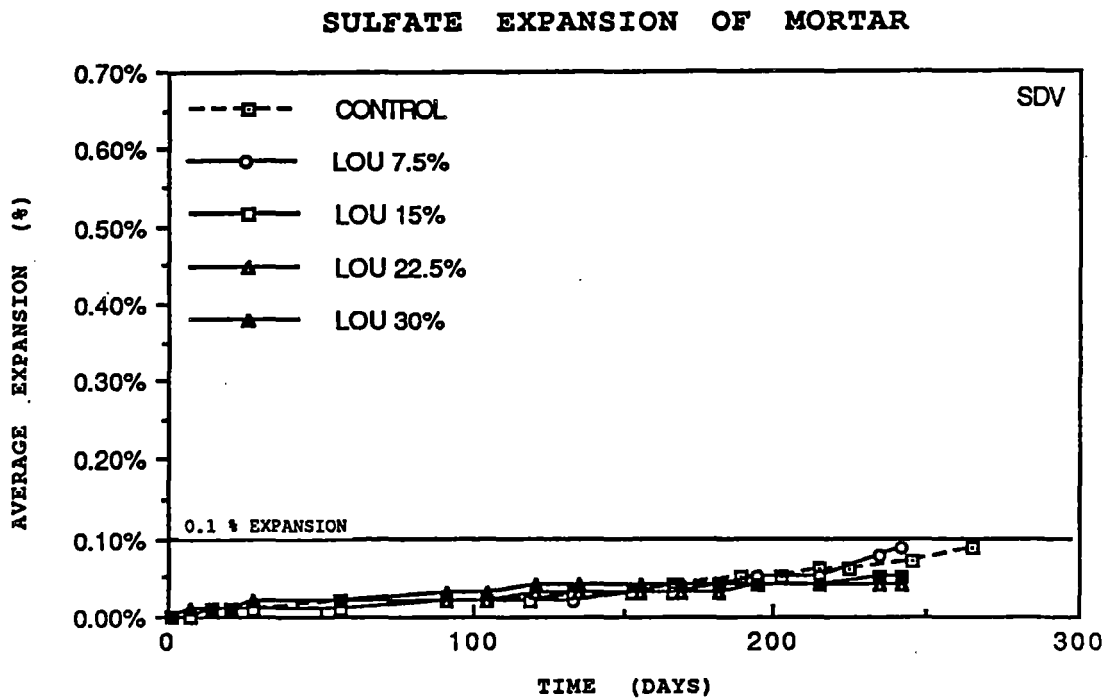
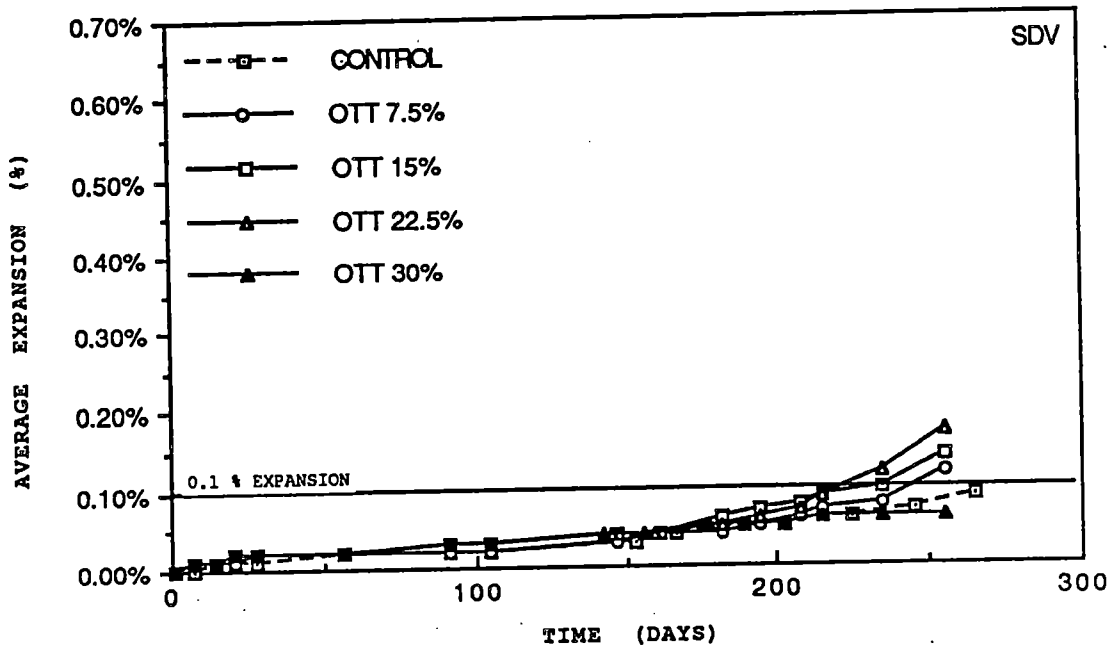


Figure 27. Average expansion versus time for mortar specimens containing Louisa fly ash and South Dakota cement.

# SULFATE EXPANSION OF MORTAR



# CHLORIDE-SULFATE EXPANSION OF MORTAR

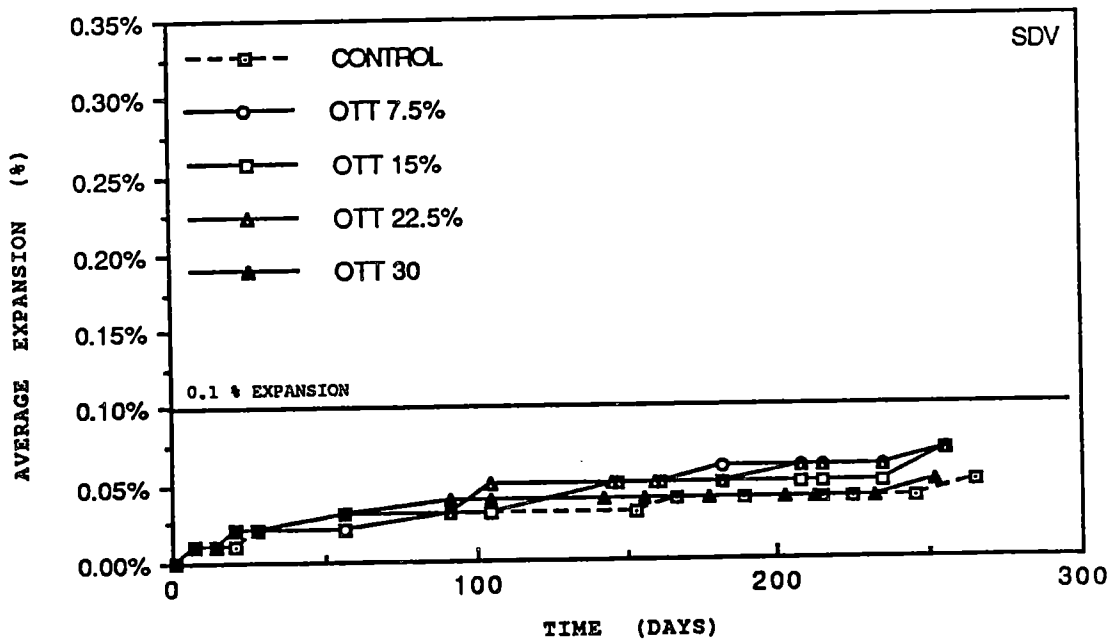
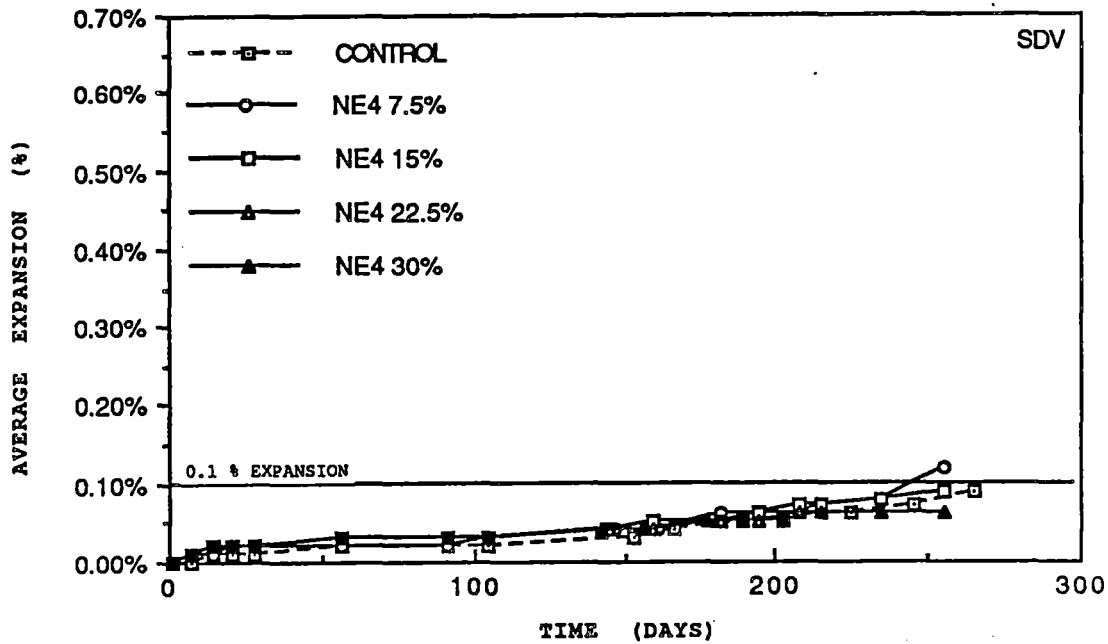


Figure 28. Average expansion versus time for mortar specimens containing Ottumwa fly ash and South Dakota cement.

### SULFATE EXPANSION OF MORTAR



### CHLORIDE-SULFATE EXPANSION OF MORTAR

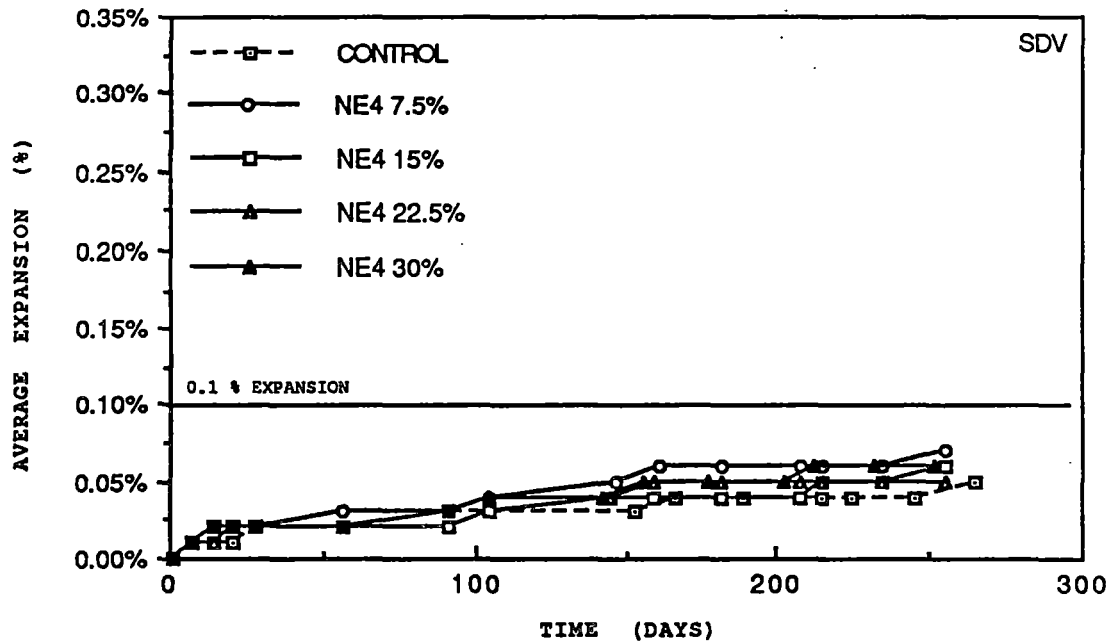
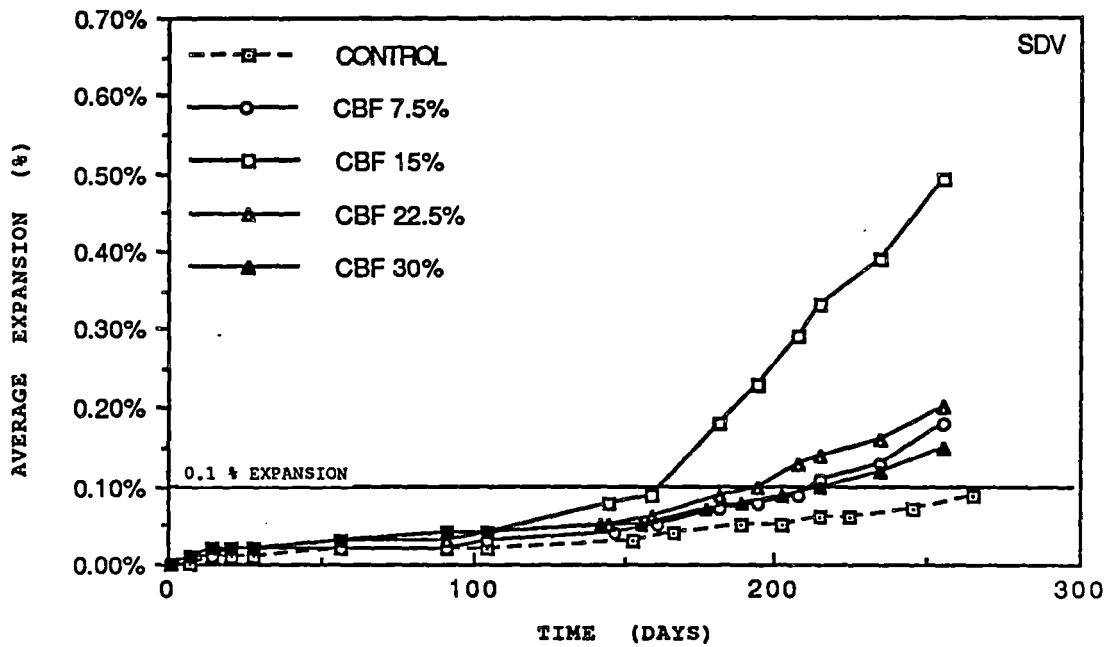


Figure 29. Average expansion versus time for mortar specimens containing Neal 4 fly ash and South Dakota cement.

## SULFATE EXPANSION OF MORTAR



## CHLORIDE-SULFATE EXPANSION OF MORTAR

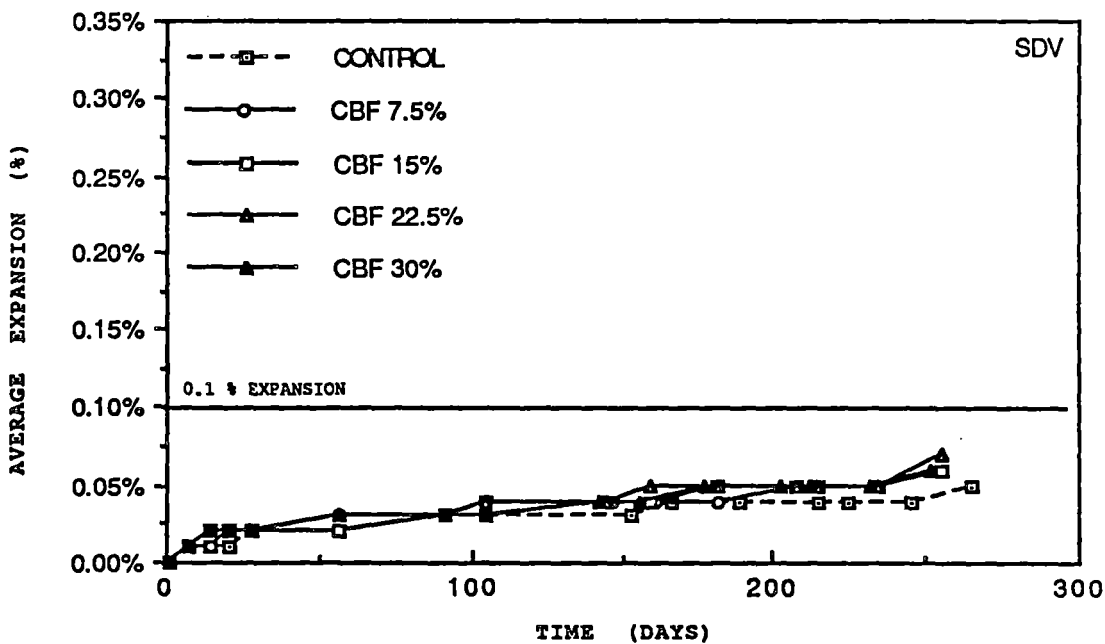


Figure 30. Average expansion versus time for mortar specimens containing Council Bluffs fly ash and South Dakota cement.

There are several key items that need to be mentioned about the various figures. First, it is readily apparent from Figure 15, that the type of cement used in the various mortar mixtures has a major influence on the expansion of the test specimens. As would be expected, the high  $C_3A$  cement (10%  $C_3A$ ) expanded most rapidly followed by the moderate  $C_3A$  cement (7%  $C_3A$ ) and then the low  $C_3A$  cement (4%  $C_3A$ ). To date, these control mortars are still intact and are submerged in the sulfate bearing solutions.

Second, fly ash type and level of replacement also appear to play important roles in the expansion of the test specimens. Typically the Clinton (Class F) and Louisa (Class C) fly ashes performed the best in the ASTM C 1012 (5%  $Na_2SO_4$ ) durability test. Their performance improved as fly ash replacement increased. Note in Figures 16, 17, 21 and 22, that by replacing 30% of either the Davenport or Dundee cement with an equivalent amount of Clinton or Louisa fly ash results in a sulfate durability performance that is comparable to Type V cement.

Specimens containing fly ashes from Neal 4 and Ottumwa generating stations exhibited erratic behavior in the sulfate durability test. Some of the Neal 4 and Ottumwa specimens performed better than the control specimens while others deteriorated rather quickly. Perhaps this suggests the presence of a pessimum amount of fly ash in these two series of mortar bar specimens.

The specimens containing Council bluffs fly ash all performed very poorly in the ASTM C 1012 sulfate durability test. In fact, the Council bluffs fly ash was the only ash that consistently caused all of the specimens to expand rapidly, regardless of which type of portland cement was used in the test specimens. Also, increasing the amount (% replacement) of Council Bluffs fly ash in the mortar mixes tended to accelerate the deterioration of the test specimens.

Surface cracking may be one reason for the rather erratic results that were observed in some of the fly ash-cement mortar specimens. Many of the test specimens containing Neal 4, Ottumwa or Council Bluffs fly ashes exhibited surface cracking after about 75 to 150 days in the sulfate bath. Once cracking was observed on the surface of the test specimens, rapid expansion generally followed. Duplicate specimens that were submerged in a lime water bath did not exhibit any expansive tendencies (maximum observed expansion was less than 0.02% at 190 days). Hence, the cracking and expansive tendencies were directly attributed to the sulfate treatment.

Since cracking drastically influences the rate of deterioration of the test specimens it may be wise to adopt some arbitrary value of expansion for comparing

the many different mortar specimens. This is the reason for placing the horizontal line at 0.1% expansion on the various figures. Typically, very few specimens exhibited much cracking at this low value of expansion.

Third, it is important to compare the behavior of test specimens placed in the sulfate bath (5%  $\text{Na}_2\text{SO}_4$ ) with those placed in the mixed salt bath (9.5%  $\text{NaCl}$  + 0.25%  $\text{Na}_2\text{SO}_4$ ). This was the reason for placing the test results in the same figure (i.e., sulfate bath results in the upper graph, mixed salt bath results in the lower graph). Presently most of the test specimens placed in the mixed salt bath are exhibiting expansive tendencies, however, in most instances the fly ash specimens are performing better (i.e., lower expansion) than the portland cement control specimens. Also, increasing the amount of fly ash in a given specimen tended to decrease the expansion of the test specimens (i.e., a dilution effect), regardless of the type of fly ash that was used. This is contrary to the results obtained from the sulfate bath. However, it is premature to make any quantitative assessments of the performance of the various portland cement-fly ash mortar specimens immersed in the mixed salt bath because of the rather low observed expansions. Additional time may be required before the mortar specimens show the true nature of sulfate attack. The specimens are currently being monitored and updated information will be available for the next report.

The results of the curing time study will not be presented in this report. The test specimens are currently submerged in the sulfate bath but additional time is required to obtain a significant response to the sulfate treatment.

A summary of the details pertaining to the concrete mixes made at the IDOT is listed in Table VII. All of the concrete mixes had air contents of  $6 \pm 1$  percent and slumps of  $2 \pm 0.5$  inches. Concrete cylinders molded from the various mixes indicated that all the concrete specimens had 28-day compressive strengths in excess of 6000 psi. Most of the mixes incorporating fly ash tended to have higher 28-day compressive strengths than the portland cement control specimens.

The preliminary results of the sulfate durability tests being conducted on a selected series of the concrete mixes are shown in Figures 31 through 34. All of the figures were constructed using the same basic format that was used for the mortar specimens. Growth (linear expansion, average of two specimens) is plotted on the Y-axis, while time (in weeks of exposure to the sulfate solution) is plotted on the x-axis. Note that the x-axis of each graph is not linear. The upper graph in each figure illustrates expansion due to a 10%  $\text{Na}_2\text{SO}_4$  solution, the lower graph illustrates expansion due to a 10% mixed salt solution (9.5%



Table VII  
Summary of Concrete Mix Parameters

Mix#	Cement	Fly Ash	%Ash	Coarse Agg	W/C	Air %	Slump (in.)	Unit wt (pcf)	28-D comp str (psi)
1	Dundee		0	Jabens	0.462	5.9	1.75	143.6	6620
2	Dundee	Ottumwa	7.5	Jabens	0.464	6.1	2.00	143.2	-
3	Dundee	Ottumwa	15	Jabens	0.439	5.9	1.75	143.2	6710
4	Dundee	Ottumwa	30	Jabens	0.413	5.8	2.25	143.6	6480
5	Dundee	Council Bluffs	7.5	Jabens	0.446	6.0	2.00	143.6	-
6	Dundee	Council Bluffs	15	Jabens	0.429	5.8	2.00	144.8	7150
7	Dundee	Council Bluffs	30	Jabens	0.413	6.3	1.75	143.2	6440
8	Dundee	Clinton	7.5	Jabens	0.464	6.1	2.25	142.0	-
9	Dundee	Clinton	15	Jabens	0.453	5.7	1.75	143.6	6590
10	Dundee	Clinton	30	Jabens	0.451	5.6	1.75	143.6	6130
11	South Dakota		0	Jabens	0.472	6.0	2.00	143.6	6490
12	South Dakota	Ottumwa	7.5	Jabens	0.470	6.0	2.25	143.2	-
13	South Dakota	Ottumwa	15	Jabens	0.453	5.9	2.00	144.0	7130
14	South Dakota	Ottumwa	30	Jabens	0.422	6.0	1.75	143.6	6990
15	South Dakota	Council Bluffs	7.5	Jabens	0.458	6.1	2.00	143.2	-
16	South Dakota	Council Bluffs	15	Jabens	0.429	5.8	1.75	144.4	6880
17	South Dakota	Council Bluffs	30	Jabens	0.418	6.5	1.75	142.8	6930
18	South Dakota	Clinton	7.5	Jabens	0.464	6.4	2.25	143.2	-
19	South Dakota	Clinton	15	Jabens	0.453	6.3	2.25	141.6	6580
20	South Dakota	Clinton	30	Jabens	0.446	5.7	2.00	142.2	6380
21	Dundee	Louisa	7.5	Jabens	0.458	6.2	1.75	143.6	-
22	Dundee	Louisa	15	Jabens	0.434	6.0	2.00	144.0	7040
23	Dundee	Louisa	30	Jabens	0.422	6.0	2.25	144.0	7230
24	South Dakota	Louisa	7.5	Jabens	0.458	5.9	2.25	143.2	-
25	South Dakota	Louisa	15	Jabens	0.448	6.0	2.25	143.2	6550
26	South Dakota	Louisa	30	Jabens	0.432	6.0	2.00	144.0	7280

Table VII continued

Mix#	Cement	Fly Ash	%Ash	Coarse Agg	W/C	Air %	Slump (in.)	Unit wt (pcf)	28-D comp str (psi)
27	Dundee		0	Lamont	0.481	6.2	2.00	143.6	6440
28	Dundee	Ottumwa	7.5	Lamont	0.452	6.6	2.00	142.8	-
29	Dundee	Ottumwa	15	Lamont	0.439	6.8	2.00	142.8	6210
30	Dundee	Ottumwa	30	Lamont	0.413	6.8	2.00	143.6	6610
31	Dundee	Council Bluffs	7.5	Lamont	0.458	6.7	2.25	142.8	-
32	Dundee	Council Bluffs	15	Lamont	0.434	6.2	2.00	144.8	7370
33	Dundee	Council Bluffs	30	Lamont	0.418	6.3	2.25	144.8	6790
34	Dundee	Clinton	7.5	Lamont	0.476	6.5	2.50	142.8	-
35	Dundee	Clinton	15	Lamont	0.458	5.9	2.00	144.8	6330
36	Dundee	Clinton	30	Lamont	0.446	5.8	2.00	144.8	6050
37	South Dakota		0	Lamont	0.462	6.0	2.00	144.0	6950
38	South Dakota	Ottumwa	7.5	Lamont	0.464	6.3	2.00	144.4	-
39	South Dakota	Ottumwa	15	Lamont	0.443	6.4	2.25	144.0	7280
40	South Dakota	Ottumwa	30	Lamont	0.423	6.1	2.00	144.8	7750
41	South Dakota	Council Bluffs	7.5	Lamont	0.458	6.4	2.00	144.0	-
42	South Dakota	Council Bluffs	15	Lamont	0.448	6.4	2.00	144.0	7050
43	South Dakota	Council Bluffs	30	Lamont	0.437	6.4	2.00	144.6	7130
44	South Dakota	Clinton	7.5	Lamont	0.464	6.4	2.25	143.6	-
45	South Dakota	Clinton	15	Lamont	0.448	6.1	1.75	144.4	6690
46	South Dakota	Clinton	30	Lamont	0.469	5.8	2.25	144.0	6690
47	Dundee	Louisa	7.5	Lamont	0.458	6.1	2.25	144.4	-
48	Dundee	Louisa	15	Lamont	0.434	6.1	2.25	145.2	7790
49	Dundee	Louisa	30	Lamont	0.423	5.7	2.25	146.0	6650
50	South Dakota	Louisa	7.5	Lamont	0.458	6.1	2.50	144.8	-
51	South Dakota	Louisa	15	Lamont	0.440	6.2	2.25	144.8	7310
52	South Dakota	Louisa	30	Lamont	0.427	6.0	2.25	145.2	7450

NaCl, 0.5% Na<sub>2</sub>SO<sub>4</sub>). The abbreviations that were used on each graph have the same meaning as they did earlier in this report. Results of the monitoring of specimen weight change and sonic modulus will not be reported here because these tests indicated only gradual increases over the initial measurements.

Only a very limited amount of information is available at the present time (a maximum of five data points) for any single specimen. Also, please keep in mind that many of the test specimens have only been exposed to sulfates for about 24 weeks (i.e., approximately 170 days). Although the sulfate test is very severe, most of the test specimens have exhibited negligible expansive tendencies (i.e., growth values less than 0.01%) during the early stages of this study. Also, remember that the IDOT C-3 concrete mix is designed to have very good durability characteristics; hence, it may take a considerable amount of time before the concrete specimens show significant response to the sulfate treatments. However, there are a few interesting features that can be pointed out in these preliminary results.

First, the control specimens expanded more than any of the other test specimens during the first 24 weeks of treatment (see Figures 31 and 34). The Dundee (Type I) cement tended to expand considerably more than the South Dakota (Type V) cement. Also, at these early stages of testing, the mixed salt solution caused roughly the same expansion as the 10% Na<sub>2</sub>SO<sub>4</sub> solution. Presently it is difficult to ascertain if coarse aggregate type has any influence on the test specimens because there appear to be some anomalous results (i.e., negative expansion or shrinkage) in some of the mixes containing Lamont aggregate. The test results appear to indicate an offset of about -0.005% in specimens containing Lamont aggregate and South Dakota cement. This minor anomaly may be due to temperature effects since the mixes were made during late September and October. This slight shrinkage will become negligible once the test specimens start to respond to the sulfate treatments.

Figures 32 and 33 illustrate the influence of Council Bluffs and Clinton fly ashes on the concrete specimens subjected to the two sulfate treatments. In general, the two fly ashes behave roughly the same because they both reduce the expansion of the concrete specimens. Also, increasing the amount of fly ash in a given mixture tended to reduce the expansion of the specimen (i.e., a dilution effect). However, as was alluded to earlier in this section, the concrete specimens are just starting to respond to the sulfate treatments; and hence, more time is required to find significant differences between the various concrete mixes.

## SULFATE EXPANSION OF CONCRETE

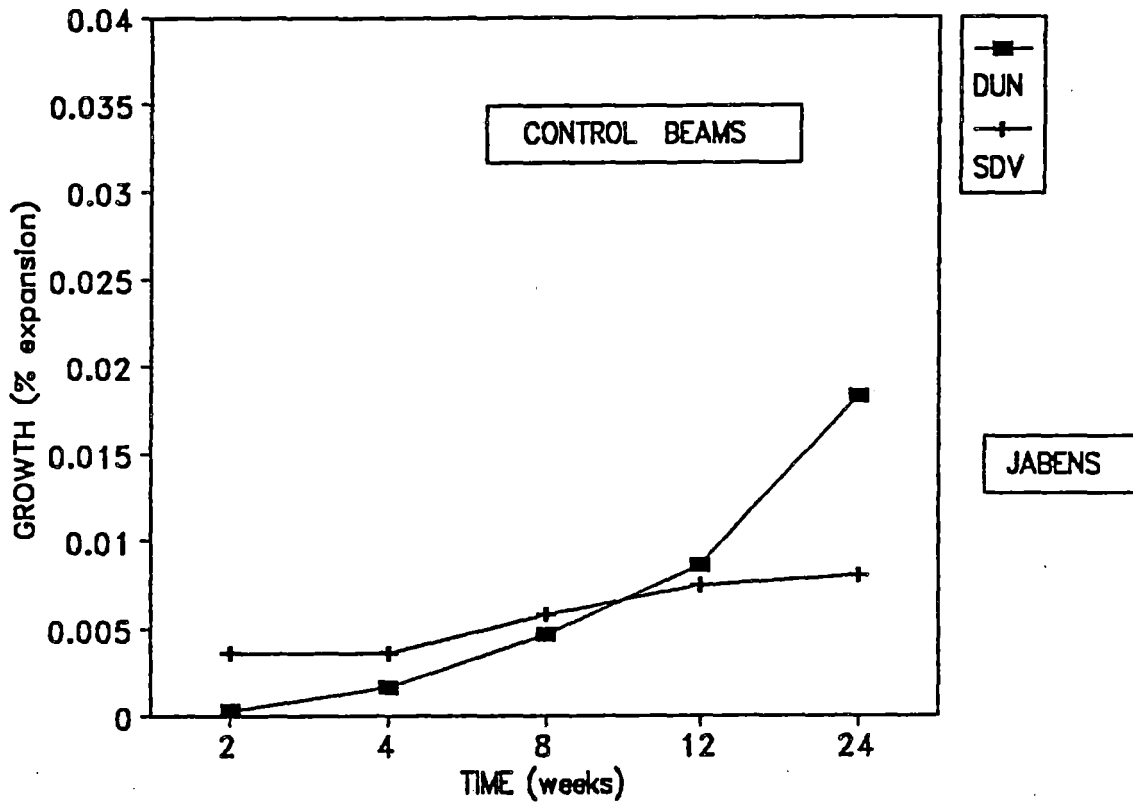
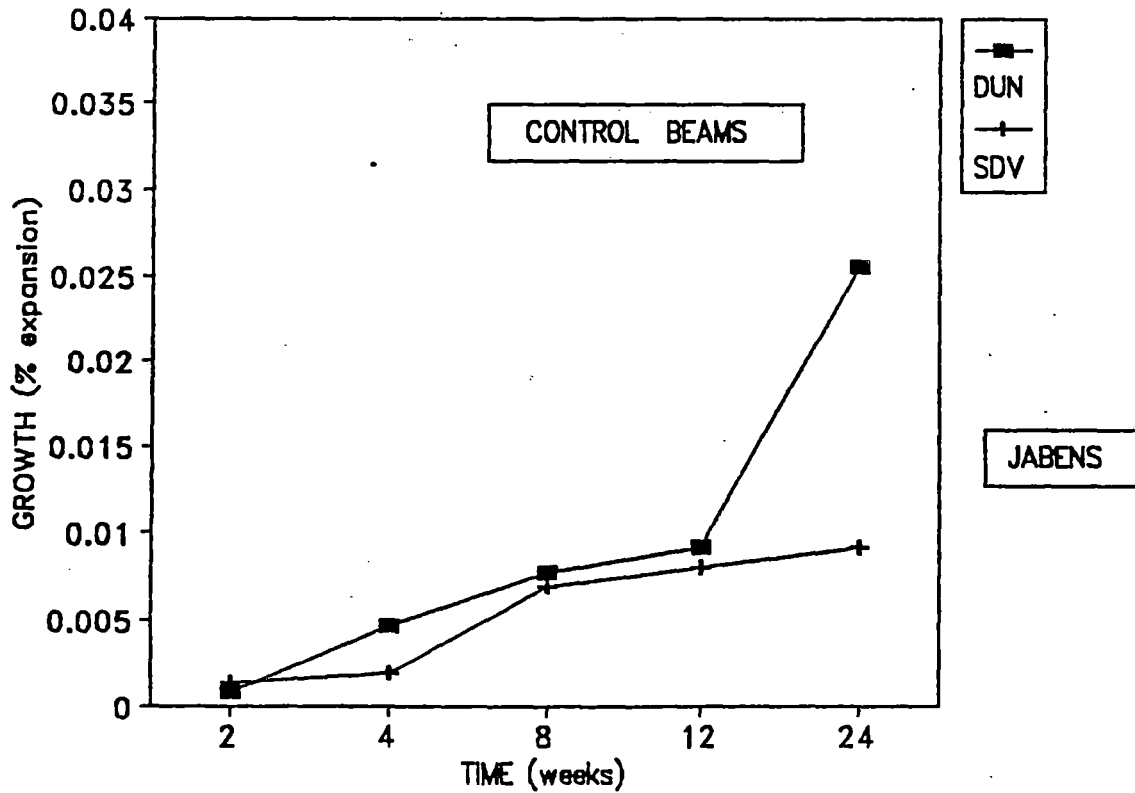
CI - SO<sub>4</sub> EXPANSION OF CONCRETE

Figure 31. Average growth versus time for concrete control, specimens containing Jabens coarse aggregate.

## SULFATE EXPANSION OF CONCRETE

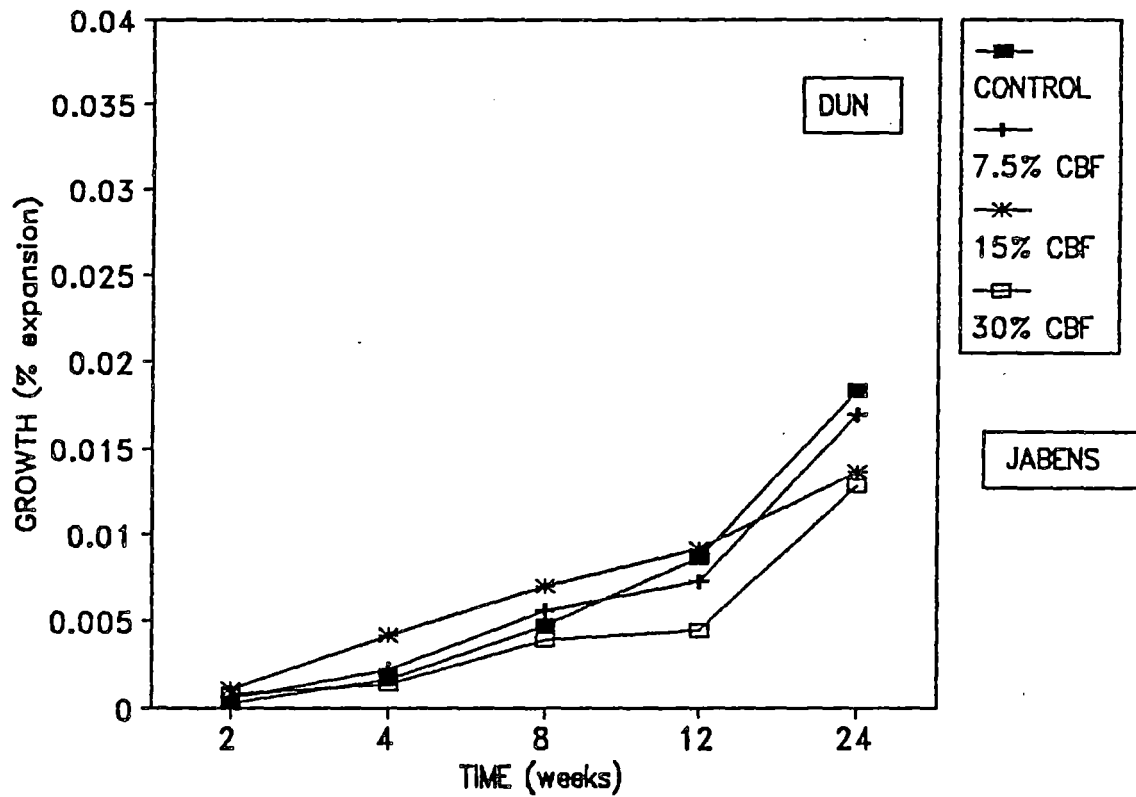
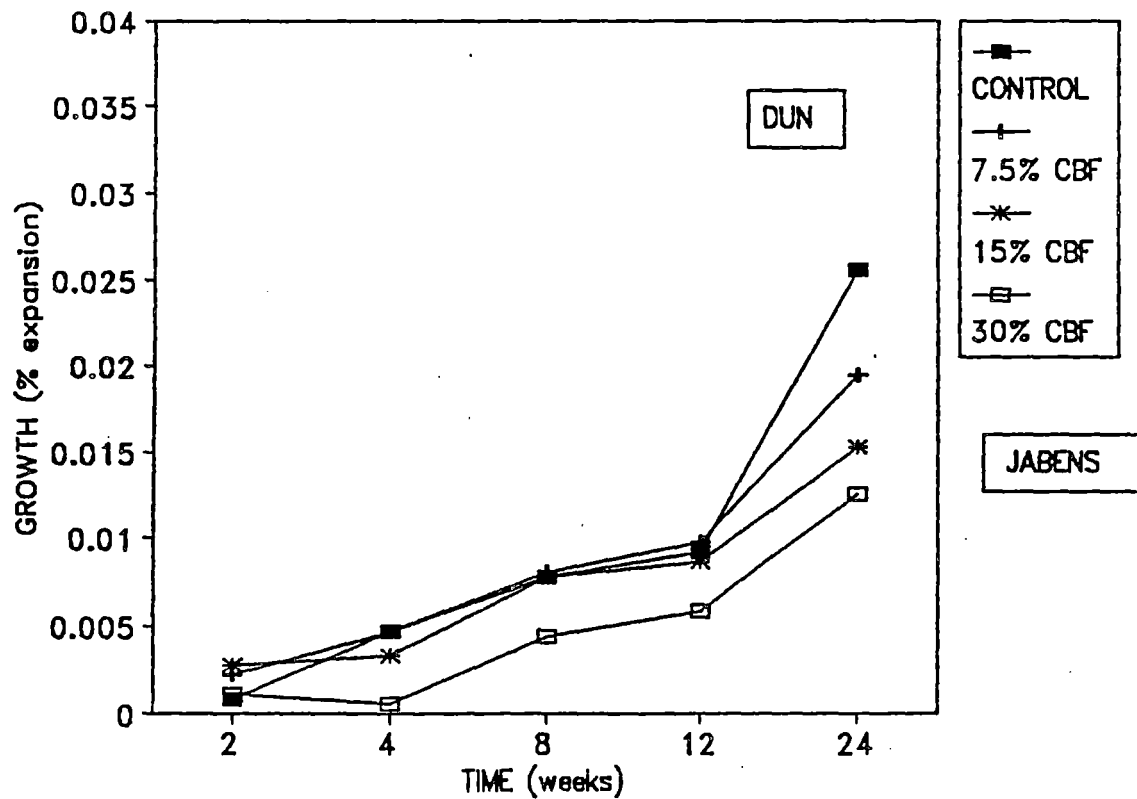
Cl - SO<sub>4</sub> EXPANSION OF CONCRETE

Figure 32. Average growth versus time for concrete specimens containing Council Bluffs fly ash, Dundee Cement and Jabens coarse aggregate.

## SULFATE EXPANSION OF CONCRETE

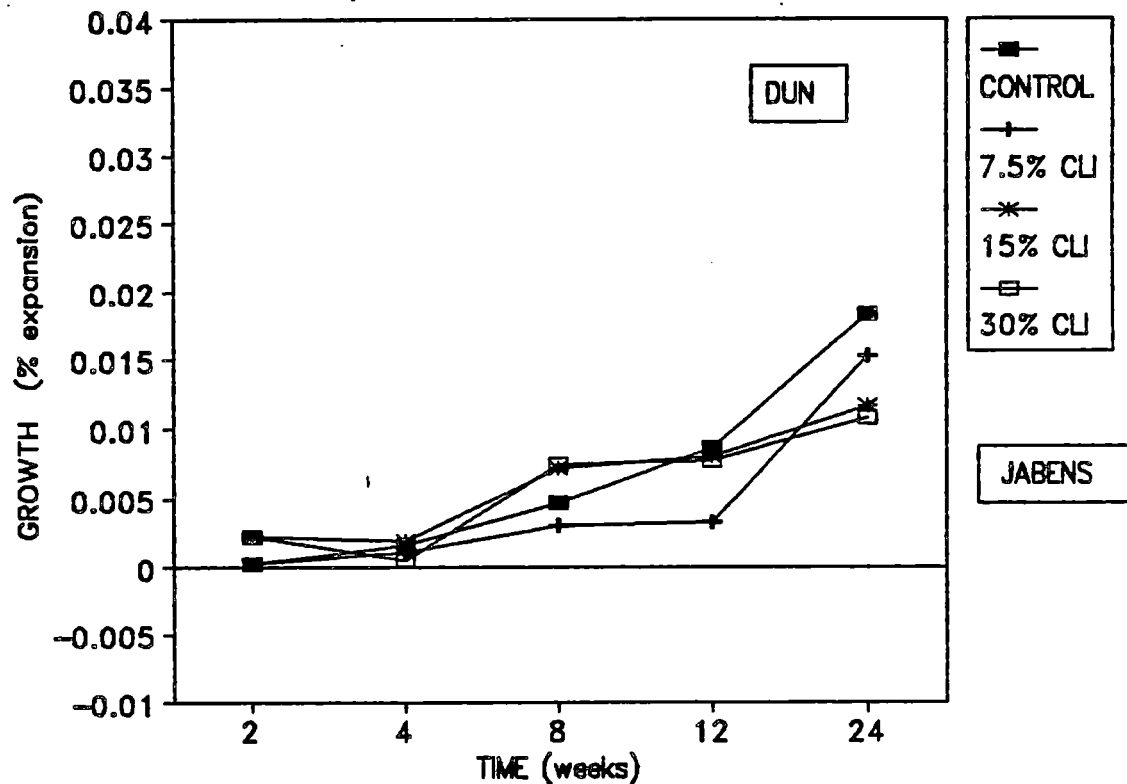
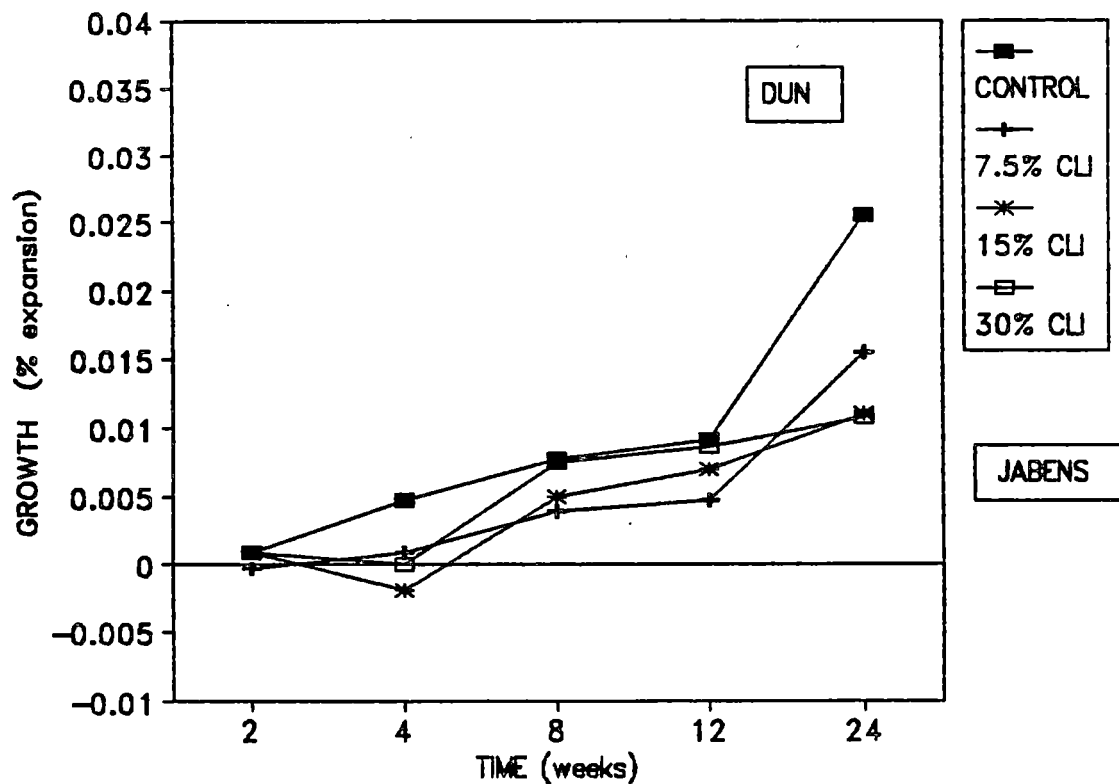
CI - SO<sub>4</sub> EXPANSION OF CONCRETE

Figure 33. Average growth versus time for concrete specimens containing Clinton fly ash, Dundee cement and Jabens coarse aggregate.

## SULFATE EXPANSION OF CONCRETE

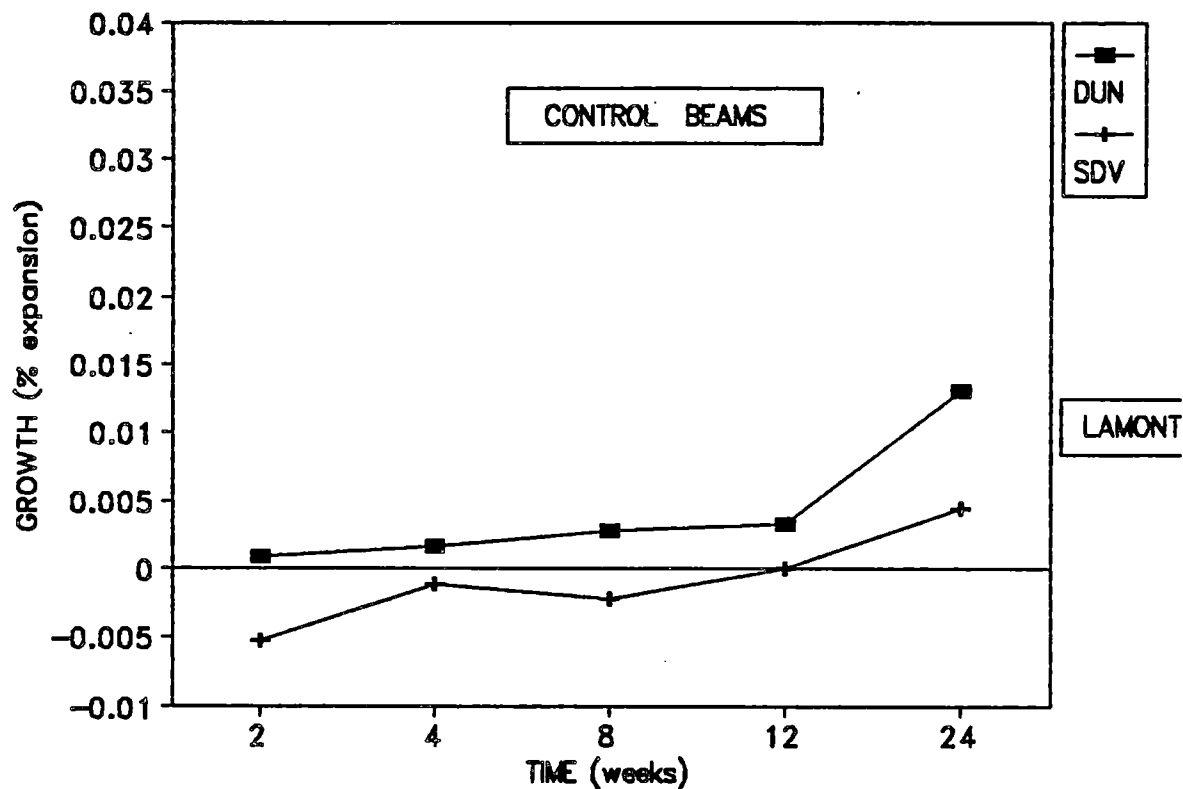
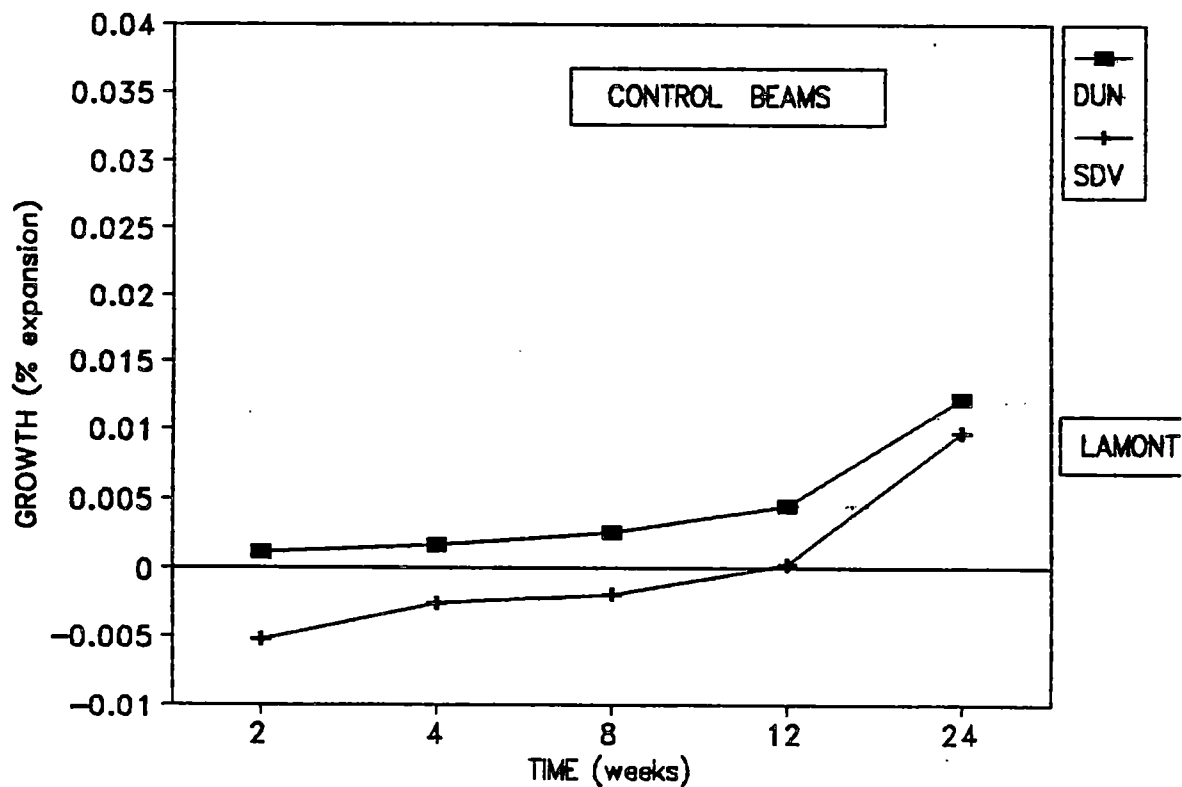
CI - SO<sub>4</sub> EXPANSION OF CONCRETE

Figure 34. Average growth versus time for concrete control specimens containing Lamont coarse aggregate.

The alkali durability specimens are still being made. To date about 60% of the work has been completed and the task should be finished in about one month. Hence, test results for the alkali durability specimens are not included in this report. Future reports will document the influence of the five fly ashes on the alkali reactivity of portland cement-fly ash mortars containing the three different fine aggregates.

#### SUMMARY AND CONCLUSIONS

In summary, the first year of this project has been spent analyzing raw materials and preparing test specimens. The preliminary paste test specimens have been molded and are currently immersed in sulfate solutions. The mortar specimens, both the primary test specimens and the curing time test specimens, have been prepared and are currently immersed in sulfate solutions. The IDOT has completed making 52 concrete mixes. These concrete specimens are currently immersed in sulfate bearing solutions. The alkali durability phase of the study is about 60% complete and should be completed in about one month. In general, the research progress is about where it was anticipated to be at the completion of the first year of the project.

Due to the relatively slow nature of sulfate attack in portland cement based materials, preliminary test results did not provide enough information to formulate firm conclusions. However, since the sulfate durability mortar specimens were made first and they have exhibited significant sulfate induced deterioration, the following preliminary conclusions can be made.

1. The 5%  $\text{Na}_2\text{SO}_4$  solution caused severe expansion in all control mortar specimens containing Dundee or Davenport cements.
2. The 10% mixed salt solution (9.5%  $\text{NaCl}$  and 0.25%  $\text{Na}_2\text{SO}_4$ ) was much less aggressive than the 5%  $\text{Na}_2\text{SO}_4$  solution. However, it did place the three control mortars in the same relative order of performance as the more severe test.
3. The relative performance of the control mortars subjected to either test solution was in agreement with the calculated concentration of  $\text{C}_3\text{A}$  in the three cements. In general, the low  $\text{C}_3\text{A}$  cement (South Dakota, Type V) performed the best, followed by the moderate  $\text{C}_3\text{A}$  cement (Davenport, Type I) and then the high  $\text{C}_3\text{A}$  cement (Dundee, Type I).



4. Fly ash type and replacement level influenced the test results of specimens placed in the 5%  $\text{Na}_2\text{SO}_4$  solution. Mortar specimens containing Clinton fly ash performed the best, followed by specimens containing Louisa fly ash, Ottumwa or Neal 4 fly ashes and then Council Bluffs fly ash. This order of performance roughly corresponds to the analytical calcium content of the various fly ashes. In general, the sulfate resistance of mortars containing Clinton or Louisa fly ash increased with increasing replacement of fly ash for cement. The sulfate resistance of mortar specimens containing Council Bluffs fly ash tended to decrease with increasing replacement of fly ash for portland cement.

#### ACKNOWLEDGEMENTS

We would like to thank all the people who helped to contribute to this project during its first year. A special thanks to IDOT personnel who have helped to procure materials and to make and monitor concrete specimens. Without their help this research project would not have been possible.

#### REFERENCES

1. Dikeou, J.T., Fly Ash Increases Resistance of Concrete to Sulfate Attack, Research Report No. 23, United States Department of the Interior, Bureau of Reclamation, 1975.
2. Von Fay, .F. and Pierce, J.S., Sulfate Resistance of Concretes with Various Fly Ashes, ASTM Standardization News, December, 1989.
3. American Society for Testing and Materials, 1990 Annual Book of ASTM Standards. Vol. 4.01, (ASTM, Philadelphia PA, 1990).
4. American Society for Testing and Materials, 1990 Annual Book of ASTM Standards. Vol 4.02, (ASTM, Philadelphia, PA, 1990).
5. Bergeson, K.L., Schlorholtz, S. and Demirel, T., Development of a Rational Characterization Method for Iowa Fly Ashes, Final Report for Iowa DOT Project HR-286, November 30, 1988.

## **APPENDIX A**

TABLE I, Appendix A  
Power plant technical details

Power Plant ----->

	Council Bluffs #3	M.L. Kapp	Louisa	Ottumwa	Port Neal #4
Boiler Type	Babcock-Wilcox	Combustion Engineering	Babcock-Wilcox	Combustion Engineering	Foster Wheeler
Maximum Generating Capacity (net MW)	700	210	650	675	600
Year on Line	1978	1967	1983	1981	1979
Fly Ash Silo Storage Capacity (tons)	4000	300	3500	3500	5000
Precipitator type	Hot-ESP	Cold-ESP	Hot-ESP	Hot-ESP	Cold-ESP
Additive used to enhance ESP performance (lbs/ton of coal)	Sodium Carbonate (1 lb/ton)	NONE	NONE	Sodium Carbonate (1 - 3 lbs/ton)	None

ESP = electrostatic precipitator

Table II, Appendix A  
Power plant operating details

Power Plant ----->

	Council Bluffs #3	M.L. Kapp	Louisa	Ottumwa	Port Neal #4
Coal Source (mine)	PRB, Wyoming (Eagle Butte-Bell Ayr)	Southern Illinois	PRB, Wyoming (Cordero)	PRB, Wyoming (Cordero)	PRB, Wyoming (Caballo)
Date when current coal contract expires	Dec. 31, 1997	Dec. 31, 1992	Dec. 31, 2002	Approx. 2000	Dec. 31, 1998
Typical Maintenance Cycle (tentative 1990)	Mid June for 2 wks; all of October	All of May; 2 wks in Oct.	Late April for 6 weeks	March thru April 2 wks in Oct.	May 18 thru June 3; 1 wk in Oct.
Start-up fuel	Fuel oil	Natural Gas	Natural gas or fuel oil	Fuel oil	Fuel oil

PRB - Powder River Basin

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## FLY ASH ANALYSIS

REPORT TO: Ken Bergeson

Date: 12/10/90

Laboratory No.: Clinton

Date Received: 6/16/90

Sample Identification: Clinton, HR-327

### CHEMICAL COMPOSITION (wt.%):

ASTM C618

Specifications

		<u>Class F</u>	<u>Class C</u>
Silicon Oxide (SiO <sub>2</sub> ).....	51.1		
Aluminum Oxide (Al <sub>2</sub> O <sub>3</sub> ).....	18.4		
Iron Oxide (Fe <sub>2</sub> O <sub>3</sub> (T)).....	14.1		
TOTAL (SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub> +Fe <sub>2</sub> O <sub>3</sub> (T))..	83.6	..70.0 min...	..50.0 min.
Sulfur Trioxide (SO <sub>3</sub> ).....	1.8	...5.0 max...	...5.0 max.
Calcium Oxide (CaO).....	5.0		
Magnesium Oxide (MgO).....	1.2		
Moisture Content.....	0.1	...3.0 max...	...3.0 max.
Loss on Ignition.....	1.0	...6.0 max...	...6.0 max.
Available Alkalies as Na <sub>2</sub> O*.....	0.9	...1.5 max...	...1.5 max.

### PHYSICAL TEST RESULTS:

Fineness			
Retained on #325 sieve, (%)...	17.0	....34 max...	....34 max.
Pozzolanic Activity Index			
With Portland Cement, (%)			
Ratio to Control @ 28 days..	93	....75 min...	....75 min.
Ratio to Control @ 7 days..	85		
Water Requirement, (% of Control)	98	...105 max...	...105 max.
Soundness			
Autoclave Expansion, (%).....	0.01	...0.8 max...	...0.8 max.
Specific Gravity.....	2.34		

### REMARKS:

\*This optional limit applies only when specifically requested.

Preliminary test results

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Approved. Scott S.

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## FLY ASH ANALYSIS

REPORT TO: Ken Bergeson

Date: 12/10/90

Laboratory No.:

Date Received: 6/16/90

Sample Identification: Louisa HR-327

### CHEMICAL COMPOSITION (wt.%):

		ASTM C618 Specifications	
		<u>Class F</u>	<u>Class C</u>
Silicon Oxide (SiO <sub>2</sub> ).....	35.9		
Aluminum Oxide (Al <sub>2</sub> O <sub>3</sub> ).....	22.8		
Iron Oxide (Fe <sub>2</sub> O <sub>3</sub> (T)).....	5.8		
TOTAL (SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub> +Fe <sub>2</sub> O <sub>3</sub> (T))..	64.5	..70.0 min...	..50.0 min.
Sulfur Trioxide (SO <sub>3</sub> ).....	1.5	...5.0 max...	...5.0 max.
Calcium Oxide (CaO).....	23.2		
Magnesium Oxide (MgO).....	4.4		
Moisture Content.....	0.0	...3.0 max...	...3.0 max.
Loss on Ignition.....	0.1	...6.0 max...	...6.0 max.
Available Alkalies as Na <sub>2</sub> O*.....	1.3	...1.5 max...	...1.5 max.

### PHYSICAL TEST RESULTS:

Fineness			
Retained on #325 sieve, (%)...	8.6	....34 max...	....34 max.
Pozzolanic Activity Index			
With Portland Cement, (%)			
Ratio to Control @ 28 days..	116	....75 min...	....75 min.
Ratio to Control @ 7 days..	107		
Water Requirement, (% of Control)	93	...105 max...	...105 max.
Soundness			
Autoclave Expansion, (%).....	0.06	...0.8 max...	...0.8 max.
Specific Gravity.....	2.60		

### REMARKS:

\*This optional limit applies only when specifically requested.

Preliminary test results

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## FLY ASH ANALYSIS

REPORT TO: Ken Bergeson

Date: 12/10/90

Laboratory No.: Ottumwa

Date Received: 6/16/90

Sample Identification: Ottumwa HR-327

### CHEMICAL COMPOSITION (wt.%):

		ASTM C618 Specifications	
		<u>Class F</u>	<u>Class C</u>
Silicon Oxide (SiO <sub>2</sub> ).....	30.9		
Aluminum Oxide (Al <sub>2</sub> O <sub>3</sub> ).....	20.2		
Iron Oxide (Fe <sub>2</sub> O <sub>3</sub> (T)).....	5.9		
TOTAL (SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub> +Fe <sub>2</sub> O <sub>3</sub> (T))..	57.0	..70.0 min...	..50.0 min.
Sulfur Trioxide (SO <sub>3</sub> ).....	2.7	...5.0 max...	...5.0 max.
Calcium Oxide (CaO).....	26.3		
Magnesium Oxide (MgO).....	4.8		
Moisture Content.....	0.0	...3.0 max...	...3.0 max.
Loss on Ignition.....	0.2	...6.0 max...	...6.0 max.
Available Alkalies as Na <sub>2</sub> O*.....	1.9	...1.5 max...	...1.5 max.

### PHYSICAL TEST RESULTS:

Fineness			
Retained on #325 sieve, (%)...	11.8	....34 max...	....34 max.
Pozzolan Activity Index			
With Portland Cement, (%)			
Ratio to Control @ 28 days..	107	....75 min...	....75 min.
Ratio to Control @ 7 days..	90		
Water Requirement, (% of Control)	93	...105 max...	...105 max.
Soundness			
Autoclave Expansion, (%).....	0.06	...0.8 max...	...0.8 max.
Specific Gravity.....	2.66		

### REMARKS:

\*This optional limit applies only when specifically requested.

Preliminary test results

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## FLY ASH ANALYSIS

REPORT TO: Ken Bergeson

Date: 12/10/90

Laboratory No.: Neal 4

Date Received: 6/16/90

Sample Identification: Neal 4 HR-327

### CHEMICAL COMPOSITION (wt.%):

		ASTM C618 Specifications	
		<u>Class F</u>	<u>Class C</u>
Silicon Oxide (SiO <sub>2</sub> ).....	35.1		
Aluminum Oxide (Al <sub>2</sub> O <sub>3</sub> ).....	18.0		
Iron Oxide (Fe <sub>2</sub> O <sub>3</sub> (T)).....	5.7		
TOTAL (SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub> +Fe <sub>2</sub> O <sub>3</sub> (T))..	58.6	..70.0 min...	..50.0 min.
Sulfur Trioxide (SO <sub>3</sub> ).....	3.2	...5.0 max...	...5.0 max.
Calcium Oxide (CaO).....	26.6		
Magnesium Oxide (MgO).....	4.9		
Moisture Content.....	0.1	...3.0 max...	...3.0 max.
Loss on Ignition.....	0.4	...6.0 max...	...6.0 max.
Available Alkalies as Na <sub>2</sub> O*.....	0.9	...1.5 max...	...1.5 max.

### PHYSICAL TEST RESULTS:

Fineness			
Retained on #325 sieve, (%)...	13.4	....34 max...	....34 max.
Pozzolan Activity Index			
With Portland Cement, (%)			
Ratio to Control @ 28 days..	104	....75 min...	....75 min.
Ratio to Control @ 7 days..	95		
Water Requirement, (% of Control)	91	...105 max...	...105 max.
Soundness			
Autoclave Expansion, (%).....	0.05	...0.8 max...	...0.8 max.
Specific Gravity.....	2.61		

### REMARKS:

\*This optional limit applies only when specifically requested.

Preliminary test results

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Approved: Swy S.



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## FLY ASH ANALYSIS

REPORT TO: Ken Bergeson

Date: 12/10/90

Laboratory No.: Council Bluffs

Date Received: 6/16/90

Sample Identification: Council Bluffs HR-327

### CHEMICAL COMPOSITION (wt.%):

		ASTM C618 Specifications	
		<u>Class F</u>	<u>Class C</u>
Silicon Oxide (SiO <sub>2</sub> ).....	30.8		
Aluminum Oxide (Al <sub>2</sub> O <sub>3</sub> ).....	16.9		
Iron Oxide (Fe <sub>2</sub> O <sub>3</sub> (T)).....	6.9		
TOTAL (SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub> +Fe <sub>2</sub> O <sub>3</sub> (T))..	54.6	..70.0 min...	..50.0 min.
Sulfur Trioxide (SO <sub>3</sub> ).....	4.0	...5.0 max...	...5.0 max.
Calcium Oxide (CaO).....	29.1		
Magnesium Oxide (MgO).....	6.6		
Moisture Content.....	0.0	...3.0 max...	...3.0 max.
Loss on Ignition.....	0.3	...6.0 max...	...6.0 max.
Available Alkalies as Na <sub>2</sub> O*.....	1.6	...1.5 max...	...1.5 max.

### PHYSICAL TEST RESULTS:

Fineness			
Retained on #325 sieve, (%)...	12.9	....34 max...	....34 max.
Pozzolanic Activity Index			
With Portland Cement, (%)			
Ratio to Control @ 28 days..	97	....75 min...	....75 min.
Ratio to Control @ 7 days..	87		
Water Requirement, (% of Control)	92	...105 max...	...105 max.
Soundness			
Autoclave Expansion, (%).....	0.11	...0.8 max...	...0.8 max.
Specific Gravity.....	2.73		

### REMARKS:

\*This optional limit applies only when specifically requested.

Preliminary test results

Materials Analysis & Research Laboratory - Participants in the Cement & Concrete Reference Laboratory cement and pozzolan testing programs.

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